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AN IMPROVED HEATING AND CIRCULATING SYSTEM TO USE IN DOUBLE-VARIATION PROCEDURE*

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ABSTRACT

A small heating unit and circulating system has been constructed for use with the double-variation procedure for refractive index determination. It furnishes water, thermostatically controlled to any desired temperature, to the microscope stage and refractometer, and can be used when only a limited supply of hot water is available. The use of water free from air prevents the formation of bubbles in the water cell of the microscope stage.

INTRODUCTION

Following Emmons'¹ specifications for the equipment necessary for using the double variation procedure for refractive index determination, the apparatus was obtained for installation in the Harvard Mineralogical Laboratories. Since there was no source of continuous hot water in the building, a fifteen-gallon electric hot water heater was purchased. When the equipment was eventually put in working order and used continuously for an hour, it was discovered that the hot water supply was inadequate to maintain a temperature above 30° C. It was, thus, necessary to either buy a much larger heater or devise a system in which a small volume of hot water could be circulated. The latter course was decided upon and the heating and circulating system described herein was the result.

It seemed desirable to incorporate in any unit the following features: (1) It should be of small volume so that the time required to change the temperature of the system would be reduced to a minimum, and so that only a small amount of additional heat would be required to maintain a required temperature. (2) It should be possible to vary the temperature either upward or downward easily and rapidly. (3) It should be controlled thermostatically so that the temperature could be brought to any pre-

* Contribution from the Department of Mineralogy and Petrography, Harvard University, No. 284.

¹ Emmons, R. C., *Am. Mineral.*, **14**, 415 (1929).

Emmons, R. C., *The Universal Stage: Geol. Soc. Am., Memoir 8*, Chap. 5 (1943).

determined value and maintained there indefinitely. (4) The water in the circulating system should be distilled to prevent deposition on the glass plates of the water cell. It should be boiled to drive off the air, preventing the accumulation of air bubbles.

HEATING AND COOLING UNIT

After several changes in design, the unit illustrated diagrammatically in Fig. 1 was constructed. It embodies most of the required desirable features. As can be seen from the diagram it is comparatively simple in design and construction. It consists of a brass tube 4 inches in diameter and $4\frac{1}{2}$ inches long with brass plates soldered at the ends. The parts shown in Fig. 1 are as follows:

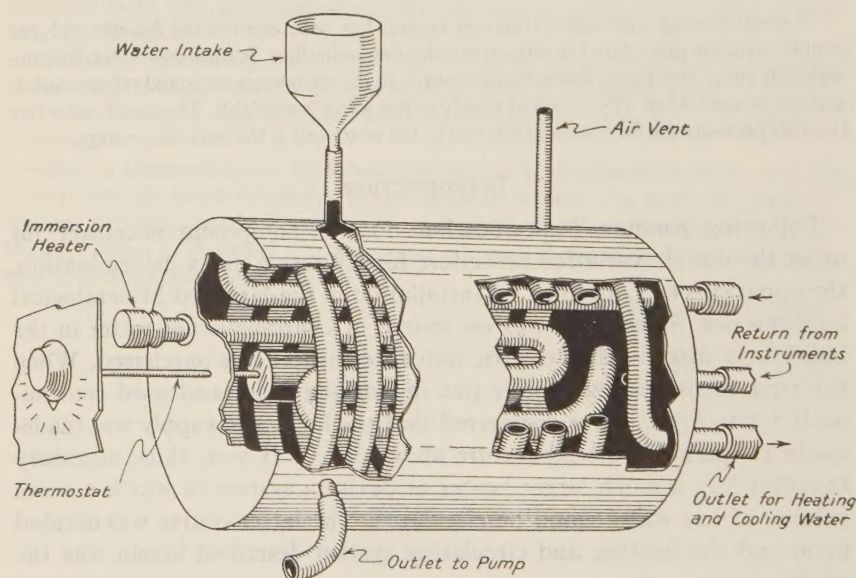


FIG. 1. Schematic diagram of heating and cooling unit.

At the center of the cylinder is a thermostat with a control rod extending through the left end. By turning the knob the thermostat can be adjusted for any desired temperature. A 150 w. immersion heater with its two leads extending from the tube is controlled by the thermostat. About 10 feet of $\frac{3}{8}$ inch copper tubing is coiled about the inside of the cylinder with the ends extending from the right end. The upper end is connected by rubber tubing to a water supply which can at will be either hot or cold. Rubber tubing from the lower end goes to waste.

From the opening, "outlet to pump," water leaves the heating unit to

circulate through the apparatus and is returned to the heater at "return from instruments." At the top of the cylinder the funnel tube is for introducing water into the system, and the other tube is a vent for the escape of air. Both tubes are 3 inches long to allow for the expansion and contraction of the water.

CIRCULATION OF WATER

Several small pumps of standard manufacture were obtained and used to pump the water from the heating unit, circulate it through the microscope stage and refractometer, and return it to the heating unit. The one illustrated in Fig. 2 proved to be the most satisfactory. It is a diaphragm pump² designed for circulating small volumes of water. Although its capacity is small, the volume of the system is so limited (approx. 1,000 cc.) that it will circulate the water about four times a minute. This is wholly adequate for keeping the instruments at a constant temperature. Both the heating unit and pump are inclosed in a box 12"×10"×6". The thermostat knob and switches for controlling the heater and pump are at the front; the water connections are at the back.

The equipment as arranged in Fig. 3 has three dial thermometers. One of these records the temperature of the water as it leaves the circulating pump. After passing this thermometer, the stream is divided, one going to the water cell of the microscope stage and the other to the refractometer. The other thermometers record separately the temperatures of the water after it leaves these two instruments and before it returns to the heating unit. Having the instruments in parallel rather than in series enables them to come to equilibrium sooner.

When all proper connections are made, distilled water that has been boiled to drive off the air is poured through the funnel tube into the circulating system. With the pump in operation the water quickly displaces the air and should be permitted to rise a short distance above the top of the heating unit. As evaporation takes place, more water should be added to maintain this approximate level. If this is done, the free water surface is extremely small and the chances of adsorbing air are reduced to a minimum.

OPERATION OF THE HEATING UNIT

In making a refractive index determination by the double variation procedure, the first measurements can be made at room temperature, which might be 23° C. If one then wishes to elevate the temperature to 35° C., the circulating pump and the immersion heater should be turned

² Marco Water Pump, manufactured by Marco Products, Bloomfield, N. J.

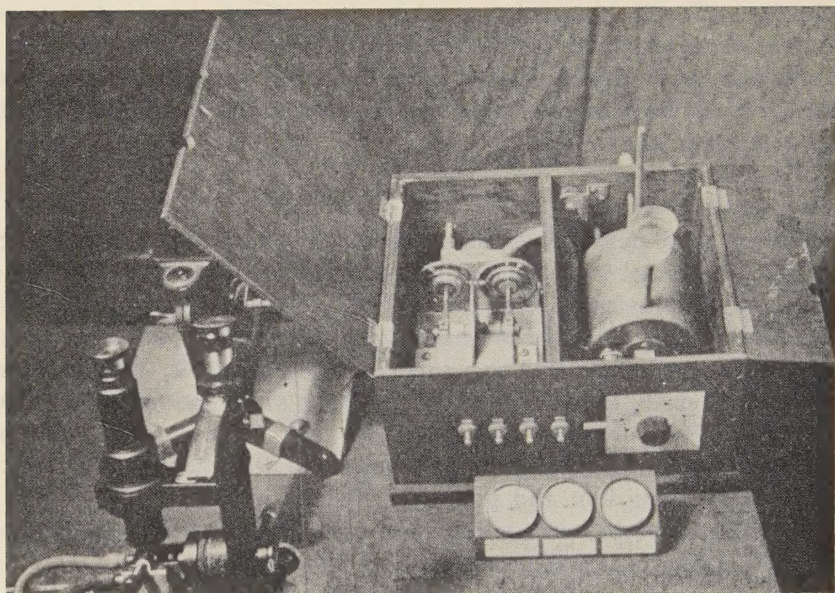


FIG. 2. Heating unit and pump.

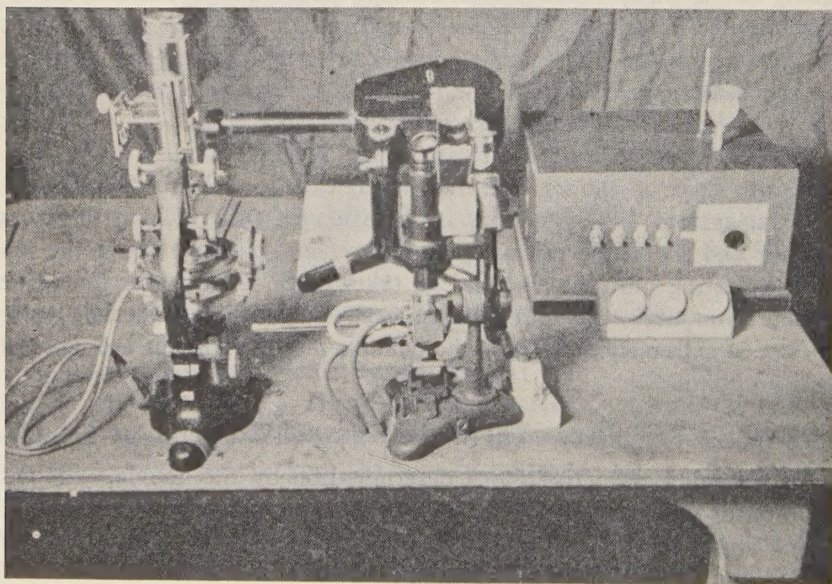


FIG. 3. Relation of heating unit to microscope and refractometer.

on, and the thermostat set for 35°C. With the 150 w. immersion heater the time necessary to raise the system the required 12° would be about eight minutes. However, if hot water is available, it should be passed through the copper coil which is completely independent of the circulating system. A limited supply of hot water if at a temperature of 70° C. will in less than a minute raise the temperature of the system close to the desired 35° C. One should watch closely the thermometer recording the temperature of the water as it leaves the pump so that when it approaches the required temperature, the hot water can be turned off. The immersion heater will then quickly add the necessary degree or two and from then on hold it at the predetermined temperature. A pilot light indicates when the immersion heater is turned on and off by the thermostat.

If there is no external source of hot water, a large immersion heater can be used to speed the heating.

When one wishes to go from a high to lower temperature, the thermostat is reset and cold water is run through the copper coil. To obtain temperatures between the temperature of the tap water and the room, a small amount of cold water should be run continuously through the copper coil. This cooling effect will be offset by the immersion heater so that the required temperature can be maintained.

CONCLUSIONS

The heating and circulating system as described has been in use in the Harvard Mineralogical Laboratories for over five years. During that time it has given excellent service and none of the parts has had to be repaired or replaced.

Although the construction was undertaken to overcome an inadequate supply of hot water, the system's advantages extend beyond this. The fact that the water is air free is in itself sufficient reason for its use, for one is not troubled with annoying air bubbles which otherwise form in the water cell on the microscope stage. In addition, it is advantageous to know that the temperature will remain constant during a series of measurements and not be subject to variations in the temperature of the hot or cold water sources.

ACKNOWLEDGMENTS

A grant from the Permanent Science Fund of the American Academy of Arts and Sciences was made to the writer to further the investigation and construction of a heat and light source to be used in connection with the double variation procedure. This grant is most gratefully acknowledged, but the work is not completed on the light source. It is hoped

that within a few months a light source with a continuous variation of wave length using absorption filters will be available.

Acknowledgment and thanks are due Mr. Donald Henderson for installing and arranging the apparatus as shown in Fig. 3.

DIFFERENTIAL THERMAL CURVES OF PREPARED MIXTURES OF CLAY MINERALS*

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ABSTRACT

This paper presents differential thermal curves of clay minerals, of prepared mixtures of such minerals, and of some clay minerals diluted with inert materials.

Variations in the size and perfection of crystallinity of particles of kaolinite appear to be reflected in variations in the intensity of the thermal reactions characteristic of the mineral.

Thermal curves of many prepared mixtures show that the thermal reactions characteristic of the individual component minerals are not always discernible, particularly if the mixing is very intimate and if the components are poorly crystallized. Caution must be used in the identification of the clay minerals in mixtures, and quantitative evaluations are very difficult.

INTRODUCTION

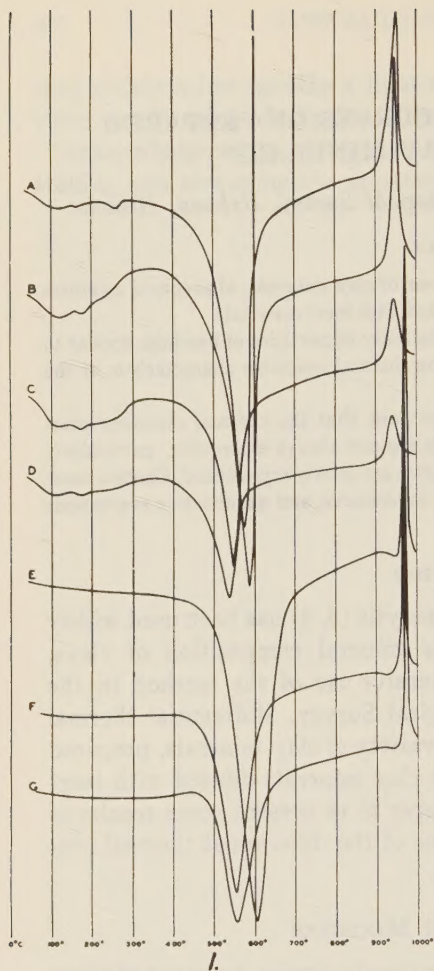
The differential thermal method of analysis (3, 4) has been used widely and successfully to determine the clay mineral composition of clays, soils, and shales. In the course of extensive use of the method in the laboratory of the Illinois State Geological Survey, differential thermal analyses have been obtained for a wide variety of clay minerals, prepared mixtures of clay minerals, and various clay minerals diluted with inert materials. The object of the present paper is to present some results of this work which are pertinent in the use of the differential thermal procedure for clay mineral identifications.

KAOLINITE—INERT MIXTURES

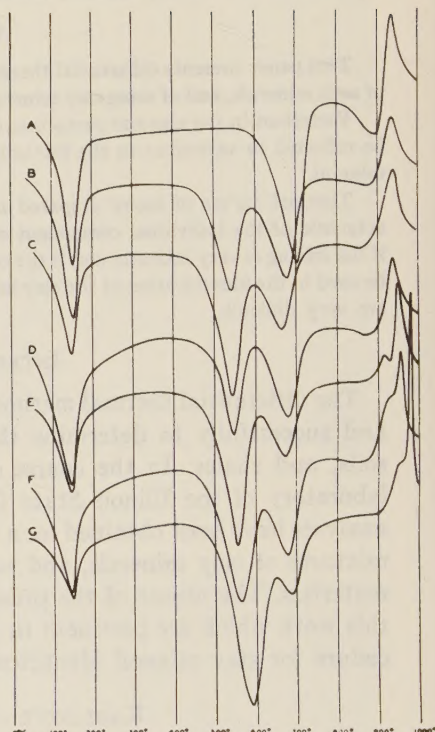
In Fig. 1, curves *A*, *B*, and *E* are for closely similar amounts of three different kaolinites (K_1 , K_2 , K_3), whose monomineral identity has been checked by x-ray diffraction analyses. The kaolinites show significant differences, namely: (1) K_1 and K_2 show broad low-intensity initial endothermic reactions whereas K_3 shows no such reaction; (2) the intensity of the large endothermic reaction with a peak at about 600° C. is slightly greater for K_3 than for K_1 or K_2 ; (3) the intensity of the final exothermic reaction is much greater for K_3 than for either K_1 or K_2 ; and (4) the final exothermic reaction in K_3 is immediately preceded by a slight endothermic reaction whereas no such reaction is shown for K_1 or K_2 .

By way of explanation for the foregoing differences in the kaolinite curves, it should be noted that kaolinites K_1 and K_2 have a lower degree

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FIG. 1. Differential Thermal Curves

- A. Kaolinite, K₁, Vera, Washington, 0.522 g.
- B. Kaolinite, K₂, Anna, Illinois, 0.460 g.
- C. Kaolinite, K₂, Anna, Illinois, 0.370 g.
- D. Kaolinite, K₂, Anna, Illinois, 0.215 g.
- E. Kaolinite, K₃, Dry Branch, Georgia, 0.427 g.
- F. Kaolinite, K₃, Dry Branch, Georgia, 0.355 g.
- G. Kaolinite, K₃, Dry Branch, Georgia, 0.230 g.

FIG. 2. Differential Thermal Curves

- A. Sodium montmorillonite, M₁, Clay Spur, Wyoming, 0.628 g.
- B. Sodium montmorillonite, 0.536 g. and kaolinite, K₁, 0.268 g.
- C. Sodium montmorillonite, 0.425 g. and kaolinite, K₁, 0.425 g.
- D. Sodium montmorillonite, 0.518 g. and kaolinite, K₂, 0.259 g.
- E. Sodium montmorillonite, 0.424 g. and kaolinite, K₂, 0.424 g.
- F. Sodium montmorillonite, 0.514 g. and kaolinite, K₃, 0.257 g.
- G. Sodium montmorillonite, 0.408 g. and kaolinite, K₃, 0.408 g.

of crystallinity than K_3 . By lower degree of crystallinity is meant less perfect stacking of the units and perhaps distortion within the units. It is noteworthy also that kaolinites K_1 and K_2 have higher plastic and bonding properties than K_3 , characteristics that are probably related to the degree of crystallinity and which might be predicted from the thermal curves.

The differences in the final portion of the curves may be taken to mean that loss of (OH) from the kaolinite lattice for well crystallized sample K_3 is not accompanied by complete destruction of lattice which occurs with the final slight endothermic reaction at about 925–950° C., whereas in the case of the poorly crystallized samples K_1 and K_2 the kaolinite lattice is completely destroyed along with the loss of (OH).

Curves *C* and *D* are for the same kaolinite as K_2 , but the sample in the furnace was diluted with precalcined Al_2O_3 . Similarly curves *F* and *G* are for the same kaolinite as K_3 , but the sample in the furnace was also diluted with precalcined Al_2O_3 . The curves for reduced amounts of kaolinite show a proportionate reduction in the size of the thermal reactions. It is noteworthy that the final exothermic reaction for K_2 is reduced proportionately more than that for K_3 .

Curve *D* might be confused with some illite curves, whereas curve *G* for about the same amount of well crystallized kaolinite would not be so confused because of the absence of an initial endothermic reaction and the character of the final exothermic reaction. Also the curves show that an initial endothermic reaction does not always mean the presence of a clay mineral other than kaolinite.

KAOLINITE AND SODIUM MONTMORILLONITE (WYOMING BENTONITE) MIXTURES

Curve *A* in Fig. 2 is that of a sodium montmorillonite, M_1 , in Wyoming bentonite. Curves *B* and *C* are mixtures of this montmorillonite and kaolinite, K_1 of Fig. 1; curves *D* and *E* are similar mixtures with kaolinite, K_2 ; and curves *F* and *G* are similar mixtures with kaolinite, K_3 . The mixtures were prepared by mixing weighed amounts of minus 200-mesh material, then soaking in distilled water overnight, followed by 30 minutes agitation in a mechanical mixer. The mixture was then allowed to dry, soaked again in distilled water, stirred 30 minutes and dried. This procedure was repeated through four cycles.

The initial endothermal peaks of the mixtures are about proportional in size to the amount of montmorillonite in the mixture. However, this peak is slightly larger for mixtures containing kaolinite K_1 or K_2 (curves *B–E*) than for mixtures with kaolinite K_3 (curves *F–G*) because kaolinites K_1 and K_2 (unlike K_3) also have initial endothermic reactions.

The second endothermal peaks are also about proportional to the amounts of montmorillonite and kaolinite in the mixtures. In mixtures with kaolinite K_3 , because of the higher intensity of the thermal reaction for this kaolinite, the portion of the second thermal reaction due to montmorillonite appears to be reduced in size.

The final part of the curves are of considerable interest. In mixtures with kaolinite K_1 (curves *B* and *C*) the sharp final exothermic reaction for kaolinite is not obvious even in mixtures with 50 per cent kaolinite (curve *C*). It seems significant that a relatively small amount of kaolinite eliminates the third endothermic reaction for montmorillonite at about 900° C., and this may be a usable clue in recognizing the presence of kaolinite in such mixtures. In case of kaolinite K_2 , the final exothermic kaolinite peak does not show distinctly in mixtures with one-third kaolinite (curve *D*), but does show clearly when the amount of kaolinite is increased to 50 per cent (curve *E*). For mixtures with the well crystallized kaolinite, K_3 , even with as little as one-third kaolinite, the final part of the curve is a distinct combination of the montmorillonite and kaolinite curves.

The kaolinites with the lesser degree of crystallinity (K_1 and K_2) break down on agitation in water to smaller particle size than kaolinite K_3 , and as a consequence the clay minerals in mixtures with kaolinites K_1 and K_2 are probably more intimately mixed than with kaolinite K_3 . It follows that the final thermal reactions due to the formation of new phases may be misleading and must be interpreted with great caution, particularly when there is any reason to suspect a mixture of clay minerals. However, when the final reactions indicate a distinct mixture, the mixing is probably not very intimate and at least some of the components are probably well crystallized. Cailiere (1) and her colleagues have also shown recently the necessity for caution in the interpretation of the final reactions in the thermal curves of some clay minerals.

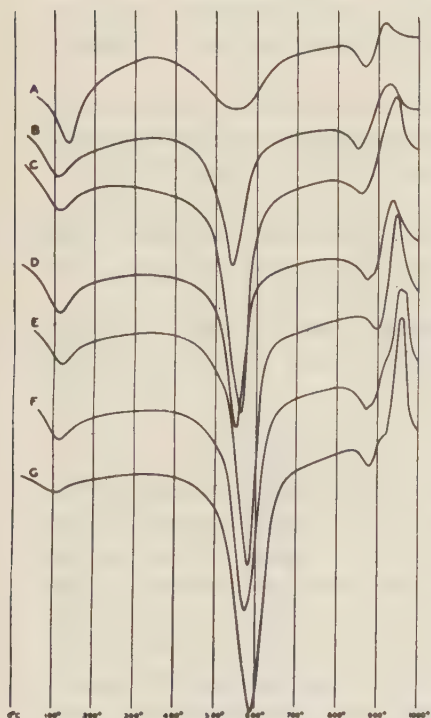
KAOLINITE AND ILLITE MIXTURES

Curve *A* in Fig. 3 is that of an illite, (2) I_1 , from an underclay of Pennsylvanian age from Grundy County, Illinois. Curves *B* and *C* represent mixtures of this illite and kaolinite K_1 ; curves *D* and *E* are for this illite plus kaolinite K_2 ; and curves *F* and *G* are for this illite plus kaolinite K_3 . The mixtures were prepared by the same procedure as used for mixtures reported in Fig. 2.

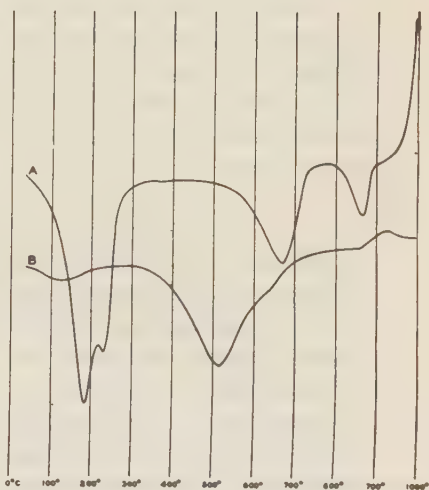
The initial endothermic peaks are roughly proportional to the amount of illite in the mixture. However, the curves (*B-E*) for mixtures with kaolinites K_1 and K_2 show slightly larger reactions than those (*F, G*) for

mixtures with kaolinite K_3 , because the former kaolinites themselves yield initial endothermic reactions.

The second endothermic peak for illite is at about the same temperature as the endothermic reaction for kaolinite. Because the intensity of this reaction is much less for illite than for kaolinite, the effect of replacing kaolinite by illite is to reduce the size of this peak. The size of this peak for a given amount of illite and kaolinite varies with the kind



3.



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FIG. 3. Differential Thermal Curves

- A. Illite, I_1 , Grundy County, Illinois, 0.504 g.
- B. Illite, I_1 , 0.504 g. and kaolinite K_1 , 0.252 g.
- C. Illite, I_1 , 0.371 g. and kaolinite K_1 , 0.371 g.
- D. Illite, I_1 , 0.518 g. and kaolinite K_2 , 0.259 g.
- E. Illite, I_1 , 0.371 g. and kaolinite K_2 , 0.371 g.
- F. Illite, I_1 , 0.502 g. and kaolinite K_3 , 0.251 g.
- G. Illite, I_1 , 0.363 g. and kaolinite K_3 , 0.363 g.

FIG. 4. Differential Thermal Curves

- A. Calcium montmorillonite, M_2 , Arizona, 0.749 g.
- B. Illite from Minford silt, I_2 , Ohio, 0.743 g.

of kaolinite. The relative amounts of kaolinite and illite could be estimated accurately only if the size of the reaction for the particular kind of kaolinite were known.

The final part of curve *B* shows that substitution of one-third of the illite with kaolinite K_1 causes little difference in the final thermal reactions for the illite. Curve *C* shows that substitution by 50 per cent kaolinite K_1 has little more effect. Unlike the mixtures with montmorillonite, the substitution of kaolinite does not tend to eliminate the third endothermic reaction for illite. It seems quite probable that as much as one-third of this kind of kaolinite might well go unidentified in mixtures of this kind.

In mixtures with one-third kaolinite K_2 , the final part of the curve does not clearly show a reaction for the kaolinite. In the mixture with 50 per cent kaolinite (curve *E*) the exothermic kaolinite reaction is evident.

In mixtures with kaolinite K_3 (curves *F* and *G*) the final part of the curve clearly shows the kaolinite reaction. The curves are distinct composites of the final part of the illite and kaolinite curves.

Apparently the final part of the curve is related to intimacy of mixing and degree of crystallinity of the kaolinite for illite mixtures as well as for montmorillonite mixtures.

MISCELLANEOUS MIXTURES OF CLAY MINERALS

Curve *A* of Fig. 4 was obtained from a calcium montmorillonite, M_2 , from a bentonite from Arizona. Curve *B* of Fig. 4 was obtained from an illite, I_2 , from the Minford silt from Ohio. Variations in the thermal curves for various members of the montmorillonite and illite groups have been presented (3).

Curve *A* in Fig. 5 is for a mixture of one-half sodium montmorillonite, M_1 , and one-half illite, I_1 , by the same wet mixing procedure. The second endothermic peak of both clay minerals shows intensities about proportional to the amount of each mineral in the mixture. The final part of the curve is very interesting in that it is that of the illite without any reflection of the montmorillonite. Again it is clear that the final portion of differential thermal curves must be interpreted with caution.

Curves *B*, *C*, and *D* of Fig. 5 are mixtures of equal amounts of illite, I_2 , and kaolinites K_1 , K_2 and K_3 respectively, prepared by the same wet mixing procedure.

The development of a double initial peak in curve for mixtures *C* and *D* is of interest and cannot be explained. An initial peak of this kind has been found in many natural clays without any satisfactory explanation.

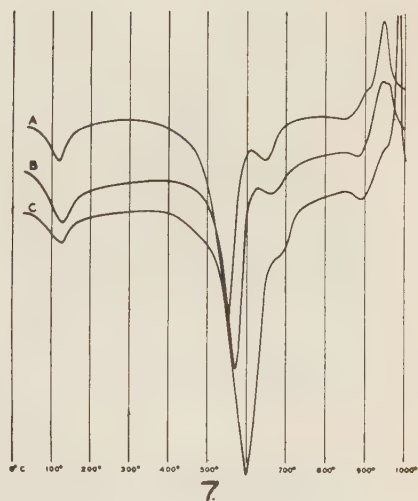
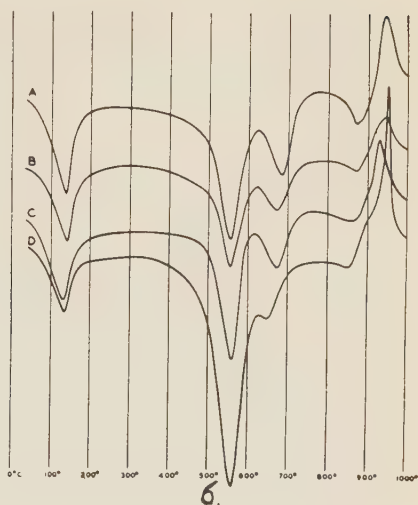
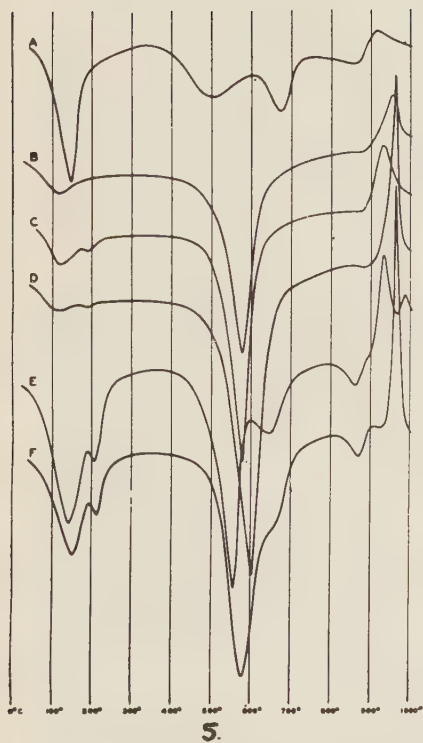


FIG. 5. Differential Thermal Curves

- A. Sodium montmorillonite, M_1 , 0.436 g. and illite, I_1 , 0.436 g.
- B. Illite, I_2 , 0.353 g. and kaolinite, K_1 , 0.353 g.
- C. Illite, I_2 , 0.350 g. and kaolinite, K_2 , 0.350 g.
- D. Illite, I_1 , 0.346 g. and kaolinite, K_3 , 0.346 g.
- E. Calcium montmorillonite, M_2 , 0.386 g. and kaolinite K_2 , 0.386 g.
- F. Calcium montmorillonite, M_2 , 0.363 g. and kaolinite K_3 , 0.363 g.

(See next page for description of figures 6 and 7.)

The second endothermic peak for the illite is completely masked by the kaolinite peak. Substitution of the 50 per cent illite for kaolinite does not affect the shape of the kaolinite peak, but does reduce the intensity of the reaction by an amount that depends on the kind of kaolinite.

The character of the final part of the curve varies with the kind of kaolinite mixed with the illite. In case of the well crystallized kaolinite K_3 (curve *D*) the kaolinite is distinctly shown whereas the illite is not. In the case of kaolinites K_1 and K_2 (curves *B* and *C*) the kaolinite is only faintly suggested. It would be easy to fail to identify the kaolinite in curves *B* and *C* purely on an evaluation of the final part of the curve.

Curves *E* and *F* of Fig. 5 are mixtures of equal amounts of calcium montmorillonite, M_2 , and kaolinites K_2 and K_3 , respectively, prepared by the same wet mixing procedure. All of the thermal reactions for the montmorillonite and for the kaolinite are shown by both curves. However, the kind of kaolinite causes distinct variations in the curve—the intensity and prominence of the montmorillonite thermal reactions are greatly reduced in mixtures with kaolinite K_3 as compared to those with kaolinite K_2 .

A comparison of the curves for the different montmorillonites and illites indicates that the kind of montmorillonite or illite also affects the character of the final peak in mixtures as well as does the kind of kaolinite.

Curve *A* of Fig. 6 represents a mixture of equal amounts of kaolinite K_1 , sodium montmorillonite, M_1 , and illite, I_1 , prepared by dry mixing. Curve *B* of Fig. 6 represents a similar mixture except that it was prepared by the wet mixing as described herein. If curve *B* were obtained for a clay of unknown composition it is likely that kaolinite would be unrecognized since it does not show in the final reactions and the endothermic reaction at about 550°C . might well be interpreted as the result of illite alone. In the curve for the dry mixture (curve *A*), the kaolinite

FIG. 6. Differential Thermal Curves.

- A. Kaolinite, K_1 , 0.223 g.; sodium montmorillonite, M_1 , 0.223 g.; illite, I_1 , 0.223 g., mixed dry
- B. Kaolinite, K_1 , 0.249 g.; sodium montmorillonite, M_1 , 0.249 g.; illite, I_1 , 0.249 g., mixed wet
- C. Kaolinite, K_2 , 0.262 g.; sodium montmorillonite, M_1 , 0.262 g.; illite, I_1 , 0.262 g., mixed wet
- D. Kaolinite, K_3 , 0.273 g.; sodium montmorillonite, M_1 , 0.273 g.; illite, I_1 , 0.273 g., mixed wet

FIG. 7. Differential Thermal Curves

- A. Kaolinite, K_1 , 0.450 g.; sodium montmorillonite, M_1 , 0.150 g.; illite, I_1 , 0.150 g.
- B. Kaolinite, K_2 , 0.468 g.; sodium montmorillonite, M_1 , 0.156 g.; illite, I_1 , 0.156 g.
- C. Kaolinite, K_3 , 0.471 g.; sodium montmorillonite, M_1 , 0.157 g.; illite, I_1 , 0.157 g.

would probably be identified since the endothermic peak at about 550° C. is rather large for illite alone and the exothermic peak at about 940° C. is suggestive of kaolinite. A comparison of curves *A* and *B* shows quite well the influence of intimacy of mixing on the thermal reactions of the clay minerals and particularly on the final reactions. When the mixing is very intimate, as much as one third kaolinite might well be missed, even though pure kaolinite gives exceedingly intense and distinct thermal reactions.

Curves *C* and *D* are for mixtures like curve *B* except kaolinite K_1 was replaced by kaolinite K_2 and K_3 respectively. Both mixtures were prepared by the wet procedure. In these curves the kaolinite would easily be spotted because of the size of the endothermic reaction at about 550° C. and because of the character of the final reactions. Kaolinite is much more distinctly shown in the curve *D* for the mixture with the well crystallized kaolinite than in curve *C*. In curves *C* and *D* the identification of illite would almost certainly be missed unless some information regarding the kind of kaolinite and therefore the intensity of the kaolinite reactions were available.

Curve *A* of Fig. 7 represents a mixture of 60 per cent kaolinite K_1 , 20 per cent of sodium montmorillonite, M_1 , and 20 per cent of illite, I_1 , prepared by the wet mixing procedure. Curves *B* and *C* of Fig. 7 represent similar mixtures except that kaolinite K_1 was replaced by kaolinite K_2 and K_3 , respectively. The presence of kaolinite is shown in each curve, but with considerable variation dependent on the kind of kaolinite. In such curves the presence of montmorillonite would be detected easily by the endothermic reaction at about 650–700° C. The presence of illite would probably be missed unless some information regarding the kind of kaolinite were available.

The writer is indebted to his colleagues Dr. W. F. Bradley for making the x-ray analyses and Mr. W. A. White for preparing the mixtures and running the thermal curves.

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GRAPHICAL DERIVATION OF REFRACTIVE INDEX ϵ FOR THE TRIGONAL CARBONATES

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ABSTRACT

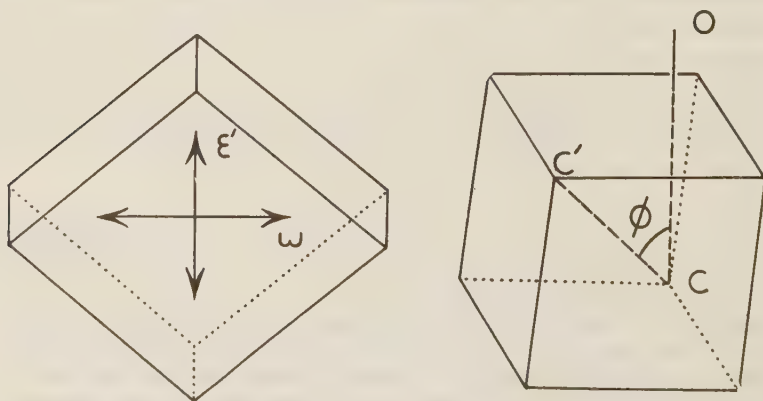
By means of a graphical method, rapid derivation of ϵ for trigonal carbonates may be obtained from the values of ω and ϵ' which are determined from cleavage fragments.

INTRODUCTION

In utilizing the immersion method to identify species of trigonal carbonates in certain industrial powders, the writer felt the need for a rapid method of determining the principal refractive indices. The presence of a perfect rhombohedral cleavage and the lack of any distinct optical indication as to when the c crystal axis is perpendicular to the optic axis of a microscope necessitates, in the determination of ϵ , a series of repeated observations of fixed or motile particles. A graph is presented to facilitate determinations carried out under routine conditions where absolute accuracy is not essential.

DERIVATION OF THE GRAPH

The principle of the method is based on the determination of the apparent refractive indices obtainable when a cleavage fragment of a trigonal carbonate rests on any one of its cleavage planes. The apparent



FIGS. 1-2. Optical and crystallographic relations in cleavage rhombohedra of trigonal carbonates. Fig. 1 (left). View of rhombohedron in direction of optic axis of microscope to show vibration-directions ω and ϵ' . Fig. 2 (right). General view of rhombohedron to show ϕ (OC , direction of optic axis of microscope; CC' , crystal axis c).

refractive indices thus obtained are ω and ϵ' (Fig. 1) (Rogers, 1923), and ϵ' is related to ϵ by the equation,

$$\epsilon' = \frac{\omega \epsilon}{\sqrt{\omega^2 \sin^2 \phi + \epsilon^2 \cos^2 \phi}}$$

where ϕ is the angle between the c crystal axis and the direction representing the optic axis of the microscope which is normal to one of the rhombohedral cleavage sets (Fig. 2) (Salomon, 1896; Johannsen, 1918; Wahlstrom, 1943). The above equation has been applied by Hutchinson (1903), Gaubert (1919), Mountain (1926) in connection with refractive index determinations by the refractometer or prism methods, but no references have been found associating the equation with a practical use of the immersion method for the trigonal carbonates generally.

TABLE 1. CRYSTALLOGRAPHIC DATA FOR THE CLEAVAGE
RHOMBOHEDRON OF TRIGONAL CARBONATES

Mineral Species	Chemical Composition	c/a (1)	α' (2)	ϕ (3)
calcite	CaCO ₃	0.8543	101°55'	44°37'
dolomite	CaMg(CO ₃) ₂	0.8322	102°38'	43°52'
rhodochrosite	MnCO ₃	0.8259	102°50'	43°39'
siderite	FeCO ₃	0.8184	103° 4'	43°23'
magnesite	MgCO ₃	0.8112	103°18'	43° 8'
sphaerocobaltite	CoCO ₃	0.809	103°23'	43° 2'
smithsonite	ZnCO ₃	0.8063	103°28'	42°57'

(1) Axial ratio values taken from Dana (1892) except for rhodochrosite, (Winchell, 1933) and sphaerocobaltite (after Baccaredda, 1932).

(2) Values of the rhombohedral angles of cleavage fragments from Bragg (1937) Ford (1917); after Dana (1892), Baccaredda (1932).

(3) Values of the angle between the c crystal axis and the rhombohedral normal obtained from the equation,

$$\sin \phi = \frac{2}{3} \frac{c}{a} \tan \frac{\alpha'}{2}.$$

These values correspond with the (0001) $\wedge$ (10 $\bar{1}$ 1) values as given by Dana (1892). The value for sphaerocobaltite is not given by Dana; that for rhodochrosite applies for an axial ratio of 0.8184.

The application of the equation necessitates a knowledge of the angle ϕ , which varies for different species of the trigonal carbonate minerals (Table 1). The largest value, for calcite, is 44°37'; the smallest, for smithsonite, is 42°57'. The average value for ϕ is therefore 43°47', and it is suggested that for the purpose set out in this paper the value be as-

sumed for all the trigonal carbonates irrespective of species. The maximum error on this assumption is ± 0.003 , which is not unreasonably high (Table 2).

TABLE 2. REFRACTIVE INDEX DATA FOR THE TRIGONAL CARBONATES

Mineral Species	ω	ϵ	ϵ'	ϵ' Calculated with True Value of ϕ	ϵ' Calculated with Assumed Value of ϕ	Difference
calcite	1.658	1.486	1.566	1.566	1.569	+0.003
dolomite	1.679	1.502	1.588	1.587	1.587	0.000
rhodochrosite	1.817	1.597	1.701	1.702	1.701	-0.001
siderite	1.875	1.633	1.747	1.748	1.746	-0.002
magnesite	1.700	1.509	1.599	1.602	1.600	-0.002
sphaerocobaltite	1.855	1.600	—	1.722	1.719	-0.003
smithsonite	1.849	1.621	—	1.732	1.729	-0.003

ω , ϵ , and ϵ' values from Winchell (1933).

A graphical representation of the relationship between ω , ϵ and ϵ' for $\phi = 43^\circ 47'$ is shown in Fig. 3. For given values of ω and ϵ' , the corresponding value of ϵ can be directly obtained from the graph to an accuracy of ± 0.001 .

By assuming the arbitrary value of $43^\circ 47'$ for ϕ , the total error incurred in the use of the graph in Fig. 3 is therefore ± 0.004 . As will be shown in the next section, this error can be substantially reduced by the application of a correction factor.

CORRECTION FACTOR

A knowledge or partial knowledge of either the chemical composition or one of the angular values of the carbonate cleavage parallelopiped (from x-ray data, for example) can be utilized to reduce the error associated with the use of the graph in Fig. 3. It is to be noted that this correction factor can be applied as a result of preliminary data obtained from the refractive indices themselves. The magnitude of error plotted against α' , ϕ , and the chemical composition (denoted by kations only) is shown in Fig. 4.* Theoretically, the error after correction is reduced to ± 0.001 .

* The straight line in Fig. 4 was derived graphically, and is sufficiently approximate to the curve obtained from the equation,

$$\Delta\epsilon' = -\frac{1}{2}\left(\frac{1}{\epsilon^2} - \frac{1}{\omega^2}\right)\epsilon'^3\Delta\phi$$

owing to the small value of $\Delta\phi$ and the approximate constancy of $(1/\epsilon^2 - 1/\omega^2)\epsilon'^3$ for the range required.

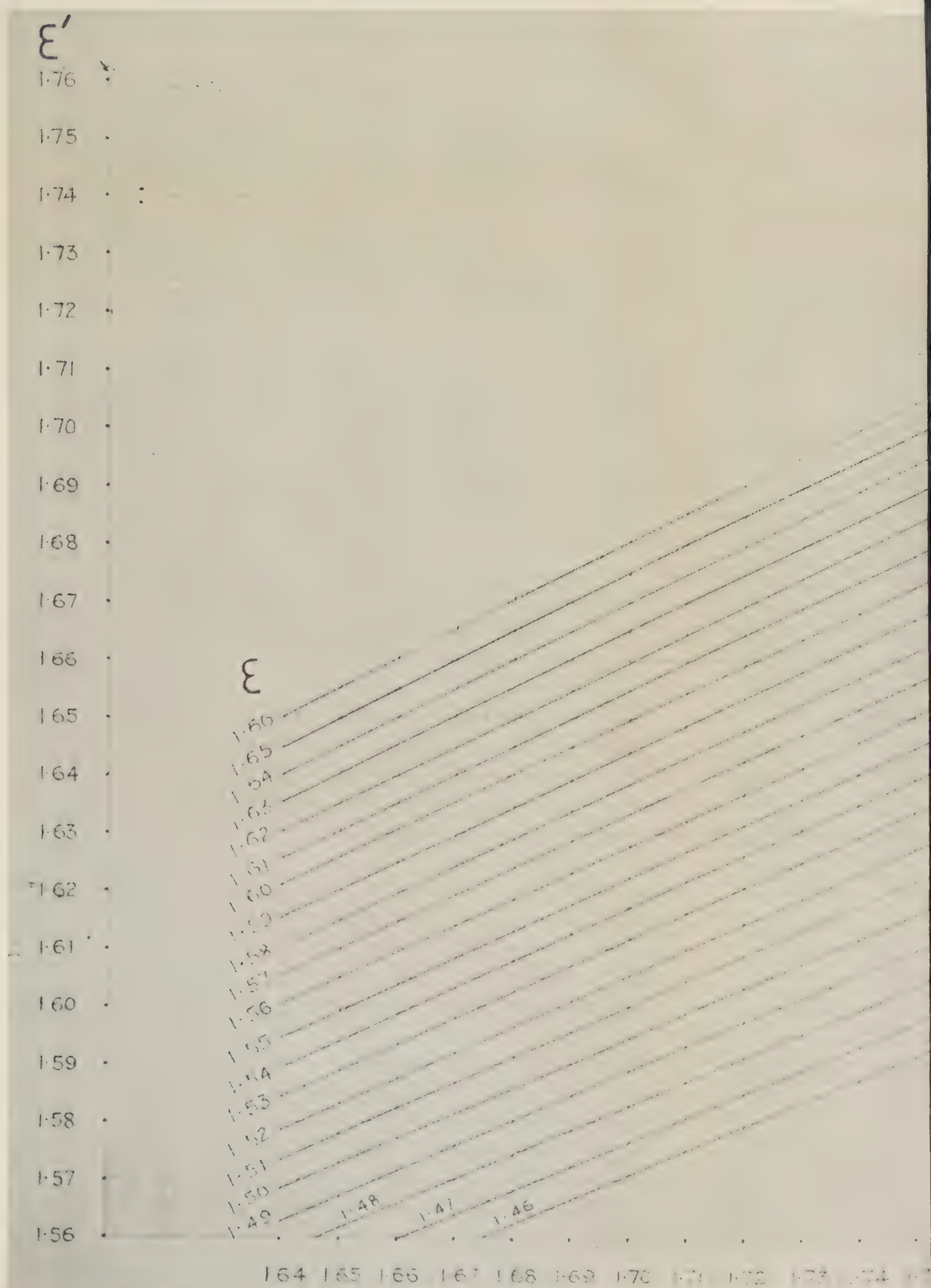
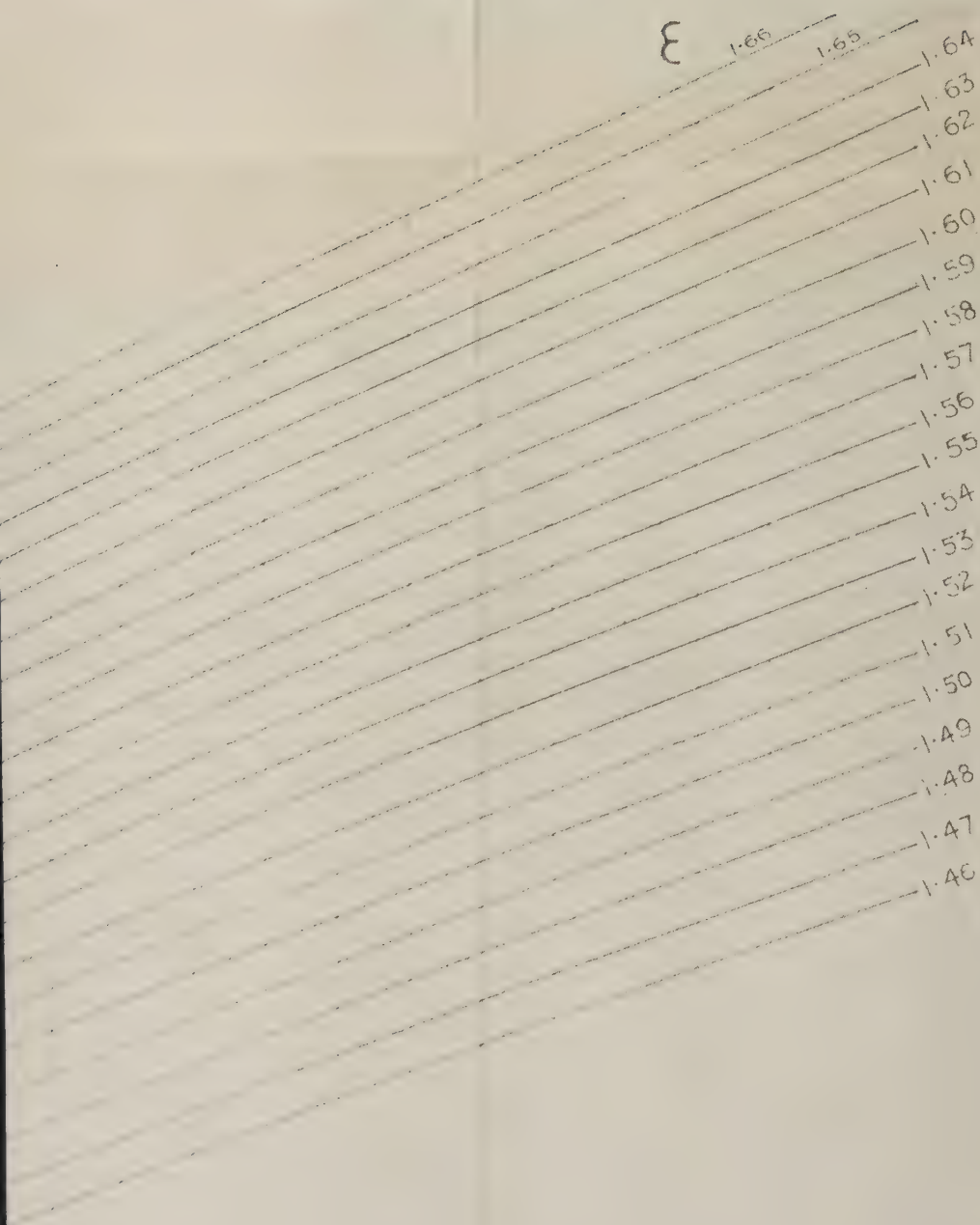


FIG. 3. Relationship between ω ,



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e, and ϵ' in trigonal carbonates.

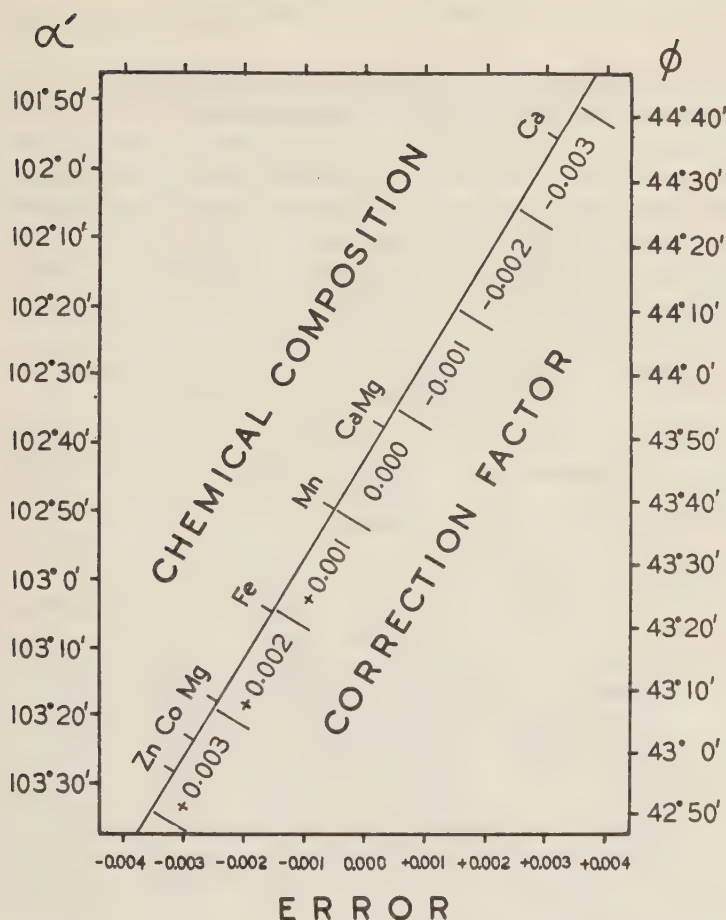


FIG. 4. Correction factor (α' , ϕ , and composition are plotted against error due to use of the assumed, instead of the true, value of ϕ).

REFRACTIVE INDEX DETERMINATIONS OF SELECTED SPECIMENS

In order to test the applicability of the graphs embodied in Figs. 3 and 4, twelve specimens of trigonal carbonate species were selected from the collections of the Geology Department, University of Bristol, for determination. Samples were crushed to small fragments, and the values of ω , ϵ , and ϵ' were determined by the immersion method. The media employed consisted of various oils and iodide mixtures; a micro-refractometer was used to determine the refractive indices of the liquids. The source of light was sodium discharge tube or suitably filtered white light, and the

temperature at which the determinations were made was maintained at $20^{\circ} \pm \frac{1}{2}^{\circ}$ C.

The results are set out in Table 3, which also includes the values of ϵ derived from the graph in Fig. 3, and the corrected values derived from Fig. 4 as a result of preliminary information of composition gained by reference to the data published by Winchell (1933). The accuracy of determination is believed to be ± 0.001 for all values except for readings above 1.8 where the accuracy is probably ± 0.002 .

Inspection of results reveals a maximum error of ± 0.004 before correction, and ± 0.002 after correction.

TABLE 3. DETERMINATIONS OF REFRACTIVE INDICES OF SELECTED SPECIMENS OF TRIGONAL CARBONATES

Specimen Number	Determinations			ϵ Read from Graph	Error in Reading of ϵ	Correction	Corrected Value of ϵ	Error after Correction
	ω	ϵ'	ϵ					
1	1.662	1.575	1.492	1.495	+0.003	-0.003	1.492	0.000
2	1.663	1.577	1.493	1.497	+0.004	-0.003	1.494	+0.001
3	1.679	1.587	1.502	1.503	+0.001	0.000	1.503	+0.001
4	1.688	1.591	1.500	1.502	+0.002	0.000	1.502	+0.002
5	1.691	1.595	1.511	1.509	-0.002	+0.002	1.511	0.000
6	1.692	1.607	1.527	1.529	+0.002	0.000	1.529	+0.002
7	1.705	1.608	1.523	1.521	-0.002	+0.002	1.523	0.000
8	1.713	1.611	1.520	1.520	0.000	+0.002	1.522	+0.002
9	1.763	1.630	1.518	1.515	-0.003	+0.001	1.516	-0.002
10	1.807	1.702	1.605	1.606	+0.001	0.000	1.606	+0.001
11	1.847	1.727	1.621	1.620	-0.001	+0.003	1.623	+0.002
12	1.872	1.745	1.634	1.632	-0.002	+0.002	1.634	0.000

1. Calcite; Styria, Austria.
2. Calcite; Mulatto, Predazzo, Italy.
3. Dolomite; West Chester, Penn., U.S.A.
4. Dolomite; Schemnitz, Hungary.
5. Dolomite; Hrubschütz, Moravia, Czechoslovakia.
6. Dolomite; Cumberland, England.
7. Magnesite, var. breunnerite; Carlsbad, Czechoslovakia.
8. Magnesite, var. mesitite; Flachau, Salzburg, Austria.
9. Ankerite; Schemnitz, Hungary.
10. Rhodochrosite; Alma, Colorado, U.S.A.
11. Smithsonite; Laurium, Greece.
12. Siderite; Siegen, Prussia, Germany.

CONCLUSIONS

1. The graphical method of deriving the value of ϵ from determined values of ω and ϵ' can probably be maintained to within ± 0.004 without correction, and to within ± 0.001 after correction. These values do not include the experimental error.

2. Use of a graphical method in deriving the value of ϵ reduces the expenditure of time and effort since careful orientative measures and repeated determinations of the refractive indices are not required. The method should be suitable for routine determinations.

3. A graphical method of deriving the value of ϵ enables a ready determination of the principal refractive indices of more than one species of trigonal carbonate in the same powder.

4. The graphs presented in this paper will be found to have other applications, as for instance in checking determinations of ω and ϵ .

ACKNOWLEDGMENT

The writer is indebted to Mr. E. Wolf for help rendered in the derivation of the graph in Fig. 3.

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QUANTITATIVE MINERAL ANALYSIS WITH A RECORDING X-RAY DIFFRACTION SPECTROMETER¹

HOWARD F. CARL²

ABSTRACT

A method of quantitative mineral analysis by means of a recording Geiger-counter x-ray spectrometer has been developed. It eliminates many of the difficulties of the photographic method and is applicable to a wide variety of mineral powders over a range of concentration from 100 per cent of the constituent being determined to a minimum of a few per cent. For quartz this minimum may be as low as one per cent. The method is particularly suited to rapid mineralogical analyses required in plant control or desired for research. It may be applied, as well, to individual samples under routine study. It is effective for slimes, coated and locked grains, and other systems difficult or impossible to identify by petrographic, spectrographic, or chemical methods. In addition the method is non-contaminating and non-destructive of the material being analyzed.

Working curves for quartz in a variety of base constituents have been derived and the logical extension to other minerals is pointed out. A detailed description of the technique developed for the use of this comparatively new instrument is given.

INTRODUCTION

X-ray diffraction has been developed in recent years into an indispensable tool in the field of mineral identification, particularly since complete commercial equipment has become more generally available. The powder method has proved especially valuable in the study of multicomponent mineral samples and those too small in particle size to permit identification by optical methods.

This technique, however, usually has been limited either to purely qualitative analyses or to quasi-quantitative estimates of the components in the more familiar mineral groupings or mixtures. Even here these estimates frequently can be reported only as above or below a certain minimum or maximum value. As the actual lower limits of detection for different minerals vary considerably, these limits are frequently uncertain.

An exception to these limitations has been the work done on the quantitative determination of quartz in mine dusts.^{3,4} In this field the nature of the sample with regard to particle size and quantity available for

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³ Clark, G. L., and Reynolds, D. H., Quantitative analysis of mine dusts, an x-ray diffraction method: *Ind. and Eng. Chem., Anal. Ed.*, **8**, 1, 39-40 (1936).

⁴ Ballard, J. W., Oshry, H. I., and Schrenk, H. H., Quantitative analysis by x-ray diffraction: *U. S. Dept. Interior, Bureau of Mines, R. I. 3520* (June 1940).

study has necessitated the development of a special technique to handle such material. This work has been broadened to include a more general analysis and the technique has been somewhat simplified and extended, by implication, to all crystalline materials.^{5,6} However, the "internal standard" method in conjunction with the use of photographic film and densitometric records thereof has been retained.

PHOTOGRAPHIC METHOD—ITS LIMITATIONS

Possibly one of the reasons that quantitative x -ray analysis has been of limited usefulness is that so many variables exist in the usual powder technique. These variables occur not only in the type of samples but also in their preparation and mounting, in the exposure and processing of the photographic film, and in the interpretation or densitometry of the patterns. Different samples often have dissimilar absorption characteristics, even with careful use of diluents; reproducible mountings of the prepared samples are difficult to achieve; differences exist in the scattering power of samples; and significant variations may occur in the total x -ray output from exposure to exposure, particularly during short exposures. All these factors tend to introduce unknown or uncontrolled variations into the final record. In addition, the complicated and often indeterminate effect of background density makes a reliable interpretation of a densitometric record of a photographic x -ray diffraction pattern even more difficult.

All of these possible variables must be considered in any technique of power diffraction analyses. The use of an internal standard eliminates some of these difficulties only partly and may be objectionable in itself, particularly if a further study of the material actually x -rayed is desired or when the time necessary to prepare numerous samples is an important consideration.

GEIGER-COUNTER METHOD

The automatic recording Geiger-counter x -ray diffraction spectrometer^{7,8,9,10} which is now commercially available* eliminates or minimizes

⁵ Gross, S. T., and Martin, D. E., Quantitative determination of crystalline materials by x -ray diffraction: *Ind. and Eng. Chem., Anal. Ed.*, **16**, 2, 95-98 (1944).

⁶ Ballard, J. W., and Schrenk, H. H., Routine quantitative analysis by x -ray diffraction: *U. S. Dept. Interior, Bureau of Mines, R. I. 3888* (June, 1946).

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⁸ Buhler, J. S., Engineering-design development of x -ray spectrometer: *Electrical Manufacturing*, **35**, No. 6 (June 1945).

⁹ Buhler, J. S., Process quality control with x -ray spectrometer: *Instrumentation* **1**, No. 6 (Nov. 1945).

¹⁰ Firth, F. G., The Geiger-Mueller x -ray spectrometer: *Colloid Chemistry* by Jerome Alexander, Vol. VI. 108-117 (1946), Reinhold Pub. Co., N. Y.

* Manufactured by the North American Philips Co., New York, N. Y.

many of the undesirable variables of the photographic method and consequently is more readily applicable to the quantitative analysis of a variety of mineral mixtures. This instrument completely eliminates the photographic record by substituting an automatically drawn curve representing intensity versus angle of x -ray diffraction. This is accomplished by recording on a standard strip-chart recorder the output of a special Geiger-counter tube which is mechanically driven through a 90° arc, the sample simultaneously being moved (at half speed) through a 45° arc. Through a collimating and filter system the counter tube receives the diffracted rays from the sample that is radiated by the x -ray beam similarly collimated, the intensity of the diffracted beam varying according to the usual Bragg relationship.

With this type of instrument the standardization of a technique for quantitative work becomes comparatively easy. The equipment, after a suitable stabilization period, provides a very steady output of x -radiation, stable operation of the Geiger-counter detection and amplification systems, and a reliable recording of the counter-tube output. The mounted sample has a comparatively large area under radiation, and the resulting diffraction may be considered to approximate a surface reflection condition which minimizes or removes many of the uncertainties due to variations in sample size, absorption, and scattering characteristics. The disadvantages of the photographic process and of the subsequent densitometric measurements are completely eliminated. A somewhat larger quantity of sample than is used in the camera technique, however, is necessary for best results. The technique of sample preparation, mounting, and examination, to be described, was developed in order to provide as reliable an analysis as possible with a minimum of effort and with the least contamination of the material to be studied.

APPLICATION OF THE GEIGER-COUNTER METHOD

One application of a rapid method of quantitative mineral analysis would be to a series of samples taken from plant operations or from a research program involving numerous tests on which a control analysis is desired. Such a method should be equally satisfactory for individual or isolated samples under routine study. The method described herein is applicable to any mineral or crystalline material having a fairly strong diffraction maximum at a Bragg angle less than 45° . It will cover a range of concentration from 100 per cent to about one to five per cent, the lower limit depending, among other factors, on the effective intensity of the strongest diffraction maximum of the crystal being analyzed. The method is rapid, non-destructive and non-contaminating, practically independent of initial particle size, locked grains, or minor surface coatings and alterations.

TECHNIQUE OF ANALYSIS

The specimen preparation consists merely of selecting a representative sample and grinding a sufficient quantity to produce about three cubic-centimeters volume of minus 325-mesh material. On difficultly comminuted material a somewhat larger particle size may be tolerated, but in any case all the material should pass a 200-mesh screen. The area of the specimen as mounted should at least equal the maximum radiated at the diffraction angle to be used for the analysis. For spectrometer angles, 2θ , down to about 10° , a surface $1" \times \frac{1}{2}"$ is used.

The sample holders are made from any convenient plastic. Such holders can be prepared by cutting $\frac{1}{8}"$ sheet material into $1\frac{1}{2}"$ squares on which are cemented $\frac{1}{4}"$ strips of thin plastic, preferably about $1/32"$ in thickness, to form an opening $1"$ by $\frac{1}{2}"$ and thereby leaving a free area for handling. Such a mount can be held readily while being loaded and can be inserted accurately into the specimen clamping device of the spectrometer.

The depth of the specimen is controlled by the thickness of the narrow strips cemented on the mount base. Where the quantity of material is limited, either a thinner mount or one having a smaller area may be used. Care must be taken, however, to prevent any reduction in the intensity of the diffracted beam because of insufficient thickness or area under radiation. When several mounts are used in quantitative work, they should be as nearly identical as possible.

The packing of the specimen into the mount is the one point in the procedure where uncontrolled variations may occur. A considerable variation exists in the facility with which different materials may be mounted. Usually minerals which are neither platy nor fibrous give little trouble. The "sticky" powders and those tending to assume a preferred orientation require special care in handling.

The surface of the specimen is formed as uniform and flat as possible and in the identical plane of the edging strips by being worked with a small, thin-bladed spatula. This procedure usually involves packing down sufficient powder to consolidate it, followed by a series of pats and sweeps to produce a uniformly smooth surface. Any curvature of this surface in a horizontal plane, as mounted, will result in an undetermined increase in the focussing, and hence higher intensity, of the diffracted rays. This step involves a certain amount of practice before the best technique of obtaining the most satisfactory surface for different types of powders has been acquired.

After the mount is prepared and positioned in the specimen holder, the spectrometer arm carrying the counter tube is set at the desired starting point. This position may be marked on the record chart, which is then synchronized with the motor-driven spectrometer arm. Subsequent angles indicated on the chart corresponding to spectrometer positions may

be determined by counting divisions from the starting point knowing the chart and spectrometer arm driving speeds.

It has been found most satisfactory to drive the scanning arm at 1 r.p.m. and the recorder chart at 20 inches per hour. This results in each division line on the chart (having 3 divisions per inch) representing 1° (2θ) on the spectrometer scanning arm. Faster scanning and recording will not always give a reliable and uniform record, and a slower operation is unnecessarily time-consuming.

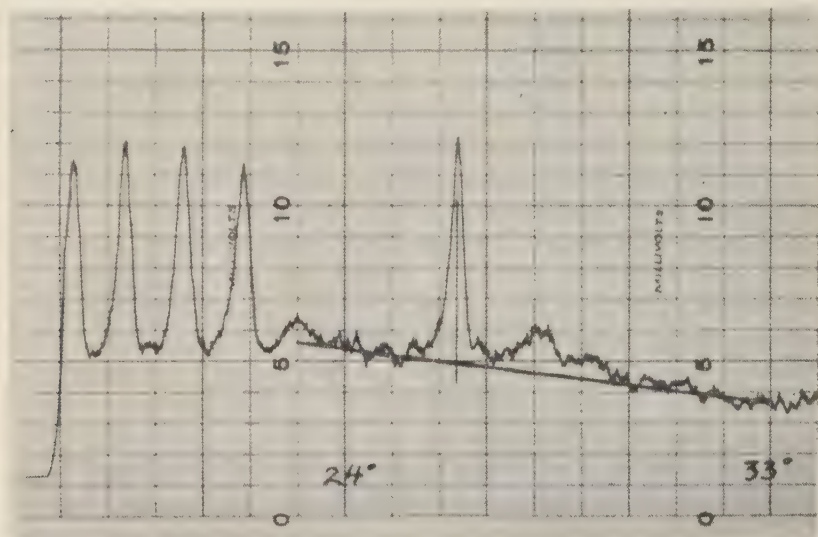


FIG. 1

A sufficient record should be obtained on both sides of the "line"¹¹ under consideration to enable a determination of the immediate background to be made. This is done by drawing a straight line connecting the average background both before and after the peak to be measured and noting the value indicated immediately under the peak. The actual value used is taken as the deflection above this background value. This is a necessary step as the background intensity will vary considerably for different materials and concentrations. However, its value may be directly subtracted from the peak value to obtain a correct index of diffraction intensity, as there is only a direct linear relation between

¹¹ The word "line" will be used to denote the recorded maximum of diffraction intensity corresponding to a diffraction line or density maximum on a film record.

intensity and recorder deflection. This numerical value of line height is, under standardized conditions, a direct measure of the actual intensity of the diffracted beam from the specimen.

Since this measurement is obtained under dynamic conditions rather than as a time-integrated determination, as is the photographic method, it is subject to certain errors resulting from the random distribution, with time, of the *x*-ray photons, as they are emitted from their source, as they are diffracted by the specimen, and as they are detected and counted by the Geiger tube. Therefore, reruns on the same sample will produce slightly varying intensity values, even with the usual amount of damping in the recording circuit. Consequently, duplicate runs are always made. It was found that an average of five determinations on the same mounted specimen could be reproduced with considerable precision. Figure 1 shows a background determination, followed by four more repeated runs to complete the record of a single mount. The duration of the background determination is unusually lengthy in this case because of superimposed faint lines.

To reduce errors that may arise from the mounting technique, five different mounts of each sample are made, and the average value of line intensity is obtained for each mount. The over-all average of these values, 25 determinations in all, is taken as the measure of the desired line height of the sample. This does not necessarily mean that enough sample for five mounts is required. A new mount may be made using all or part of the material from a previous mount. A fresh surface is what is desired.

SPECIFIC APPLICATION: QUARTZ IN VARIOUS MINERALS

Quartz was selected as the first mineral to be analyzed by this method. It occurs frequently as an impurity in other industrial minerals, and its analysis by spectrographic methods is not possible in the presence of silicates and is frequently difficult by chemical methods. When it occurs in a very fine particle size or in "locked" or coated grains a petrographic analysis is likewise difficult.

Standard samples of quartz were prepared in concentrations varying from 1 to 80 per cent in pyrex glass, BeO (bromellite), bentonite, pyrophyllite, spodumene, albite, talc, NiO (bunsenite), galena, a pyrope-almandite-type garnet, and magnetite, respectively. The minus 325-mesh materials were weighted on an analytical balance and transferred directly to a small 250-mesh screen. Each mixture was brushed through this screen at least four times thereby providing a thorough mixing of the components. It had been determined previously that this method of mixing rapidly produces a homogeneous sample. These mixtures, including a 100-per cent quartz sample, were then run on the spectrome-

ter and the corresponding heights of the 3.35A quartz line determined.

The particular instrument settings for such a quartz analysis are as follows: The x -ray beam exit slit and the counter tube defining slit are both set at "medium" width and 6 mm. height. A nickel filter is used. The recorder coupling controls (furnished with the instrument), are set at a value of 8 for sensitivity or "amplitude" and at 5 for "damping." Operation at these dial settings give practically a full scale deflection for pure quartz.

The x -ray source and counter-tube units should be given about an hour warm-up period before any quantitative measurements are made. Satisfactory operating conditions are reached when the output of the x -ray tube and the voltage on the counter tube have dropped to their steady values. The power supply of the recording unit should also be turned on for a short time, although its warm-up period does not seem to be critical.

To determine the background under the quartz peak a record is usually made for 3 or 4° on both sides of the line. A single background determination for each mount is sufficient. For duplicate runs on the same mount, scanning may be started just before the quartz line begins and continued just past the peak. A manual resetting of the scanning arm will then repeat this record.

With practice, five runs on the same mount, including a base line determination can be made in from 5 to 7 minutes and a complete determination on five mounts of a single sample can be made in about 45 minutes. The relative ease of mounting some materials may decrease the time occasionally.

WORKING CURVES

From the data obtained there has been drawn a series of curves, representing recorded height of the diffracted quartz line versus concentration of quartz for mixtures having x -ray absorption characteristics for copper radiation varying from the extremely low absorption of BeO to the high value of Fe_3O_4 and for a range of concentration from 100 to 1 per cent by weight of quartz in the base mineral. It was found that concentrations of quartz below 1 per cent could not usually be measured reliably, although in many mixtures, particularly in BeO, less than this amount could easily be detected.

In plotting these curves with a linear ordinate representing line height, it became apparent that for relatively low concentrations of quartz a logarithmic scale was not as suitable for the abscissa as a linear one. Consequently these curves were divided into two parts, from 100 to 10 per

cent quartz being plotted on a logarithmic scale, while amounts below 10 per cent were plotted on a linear scale.

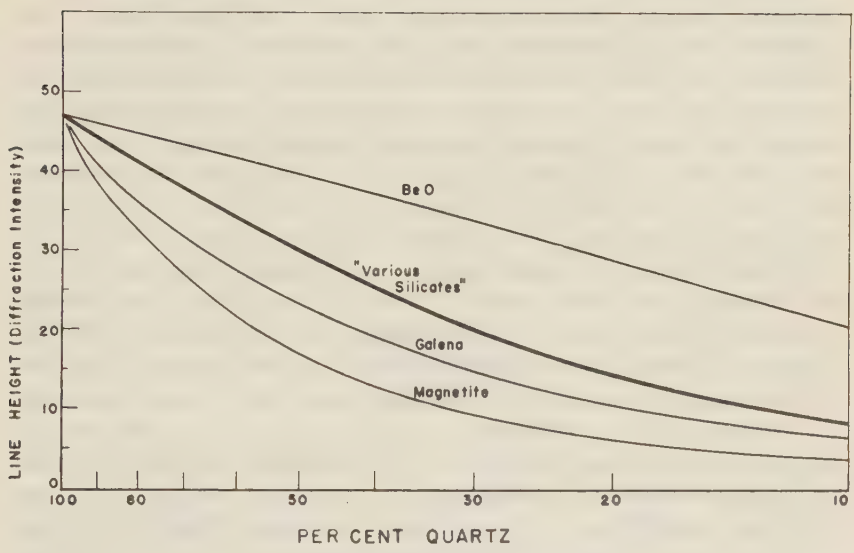


FIG. 2

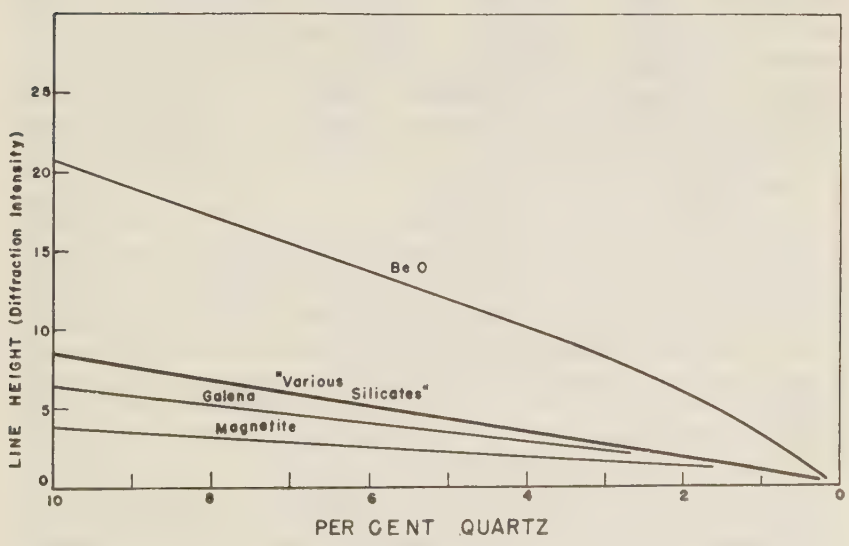


FIG. 3

The curves shown, Figs. 2 and 3, are only a few of those determined or possible within a family of curves covering the entire range of absorption characteristics exhibited by minerals. The heavy curve marked "Various Silicates" includes powdered pyrex glass, spodumene, talc, albite and bunsenite, while the bentonite clay curve is almost coincident. Although the curve for bromellite differs considerably, it probably represents the case of minimum absorption. It is certain that few minerals will have a characteristic curve between glass and BeO. Magnetite has one of the highest absorptions for copper radiation, although this value may not be the maximum possible. Its curve is much lower than that of galena or of the iron-bearing garnet studied.

Before a quantitative quartz analysis can be made on an unknown, a few representative curves similar to those shown in Figs. 2 and 3 must be obtained, using the particular apparatus and technique to be employed in future analyses. Thereafter for a quartz analysis of an individual sample or of a series of samples having similar matrix material it is only necessary to know the location of the working curve of that matrix material within the family of curves previously constructed. This can be determined by a measurement of one or two synthetic mixtures of the base material and a known amount of quartz. It might be determined merely from a knowledge of the composition of the base material. The amount of quartz to be used in a synthetic mixture should be either about 5 per cent or 25 per cent, depending on the probable range of quartz concentration in the samples to be studied. A measurement of the intensity of the quartz diffraction in these synthetic samples using the developed technique will then establish a point on the desired working curve within the family of such curves, and the complete curve may then be drawn through this point by a process of visual interpolation between the nearest bounding curves.

In practice it will be found that a large number of the more common minerals with low iron or heavy element content will possess very similar absorption characteristics and hence will have the same working curve within the limits of accuracy with which such curves may be determined. Therefore, an immediate analysis may be made on many samples of known constituents without the necessity of first running a synthetic mixture.

ACCURACY OF ANALYSIS

The fluctuations in the measurements of line height used as an index of diffraction intensity necessitate taking five readings on each mount. Likewise the possible non-uniformity of mount surfaces requires a measurement of five different mounts of the same sample. Although these in-

dividual readings may vary among themselves by a full division or more (on a scale of 50), the averages of all 25 readings can be reproduced to within about one-half division, or with a precision of about one per cent. Translated into amounts of quartz, this means that an analysis is good to within 10 per cent of the value of the quartz determined in the high range (Fig. 2), and to within plus or minus one per cent quartz in the low range (Fig. 3).

APPLICATION TO OTHER MINERALS

In general, the technique developed for the analysis of quartz should be applicable to the analyses of other minerals. For example, rutile in ilmenite was found to produce a working curve similar to quartz in magnetite with slightly different coupling control settings. Likewise magnetite in ilmenite (with iron radiation), various other opaque minerals, and many systems difficult to handle by other methods can be analyzed by this means.

DISCUSSION AND CONCLUSION

The preparation of the mounts, the one step in which completely standardized conditions do not prevail, needs further investigation. Possibly some other method of mounting samples can be developed to improve the technique and reduce the time required for an analysis. A step in this direction may be in the use of the recently described rotating sample holder¹² designed to present a greater effective surface area to the x-ray beam and to reduce the effect of preferred orientation.

The recording Geiger-counter x-ray diffraction spectrometer produces diffraction patterns more suitable for quantitative analysis of a larger variety of minerals while using the same radiation, copper $K\alpha$, than does the usual powder camera technique. It produces a permanent record which may be measured conveniently and interpreted reliably and in less time than the photographic method. For non-destructive quantitative mineral analysis on uncontaminated samples, its facility of standardization and ease of operation make it an excellent analytical tool.

ACKNOWLEDGMENT

This work was done at the Eastern Experiment Station, Bureau of Mines, under the general supervision of J. B. Zadra, Superintendent, and Chief, College Park Division, Metallurgical Branch, College Park, Md., and under the immediate supervision of Alton Gabriel, whose interest and cooperation in this problem are appreciated.

¹² Champayne, E. F., Rotating sample holder for x-ray diffraction studies: *Rev. Sci. Inst.*, **17**, No. 9, 345 (Sept. 1946).

TRIPLITE CRYSTALS FROM COLORADO¹

C. W. WOLFE² AND E. WM. HEINRICH³

ABSTRACT

The first measurable triplite crystals to be recorded were found in the Mica Lode pegmatite, Eight Mile Park, Fremont County, Colorado. Two other new triplite localities are the School Section pegmatite in the same district and another pegmatite in El Paso County, Colorado. The triplite occurs in hydrothermal replacement units along the foot-wall sides of core pods. On the basis of crystallographic measurements of four crystals and x -ray examination of an oriented section, triplite is monoclinic, prismatic $-2/m(?)$, with $a_0=11.90$, $b_0=6.48$, $c_0=9.92$, $\beta=105^\circ 53'$. The space group is $I\ 2/m?$ The optical properties and specific gravity of the Mica Lode material correspond closely with those of triplite from the 7U7 Ranch, Arizona, and it is believed that the chemical composition is also similar.

INTRODUCTION

While engaged in a study of the pegmatite deposits of Eight Mile Park, Fremont County, Colorado, the junior author obtained a number of large, rudely faced crystals of triplite. The crystallography and x -ray structure of these crystals were studied subsequently by the senior author in the Mineralogical Laboratory of Harvard University. In view of the fact that the material represents the first recorded find of triplite, crystallized sufficiently well for measurement, it is believed that a detailed description is warranted. The senior author has made all the crystallographic and x -ray measurements and calculations. Geological and paragenetic information has been supplied by the junior author. Responsibility for other parts of the paper is assumed jointly. Thanks are due to the late Dr. Charles Tozier of the Committee on Visual Education of Harvard University for the photograph of the triplite crystals.

GENERAL GEOLOGY

The geology of the Eight Mile Park pegmatite area has been studied by the junior author (Heinrich, 1947). The area is a 25-square mile plateau roughly bisected by the Royal Gorge of the Arkansas River. It is underlain by pre-Cambrian rocks of three main types: (1) Pikes Peak granite, (2) Idaho Springs schist, and (3) injection gneiss formed by lit-par-lit intrusion of schist by granitic material. The northwestern unit is granite which is bounded on the southeast by a narrow northeast-trend-

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ing belt of Idaho Springs schist. The southeastern part of the area is underlain by injection gneiss.

Moderately to steeply dipping pegmatite sills and concordant lenses are locally abundant in the Idaho Springs formation near the contacts with the Pikes Peak batholith. They attain a maximum length of 3000 feet and a thickness of 800 feet. Within the granite, also near the margins of the batholith, occur large, flat-lying, discoidal bodies of pegmatite

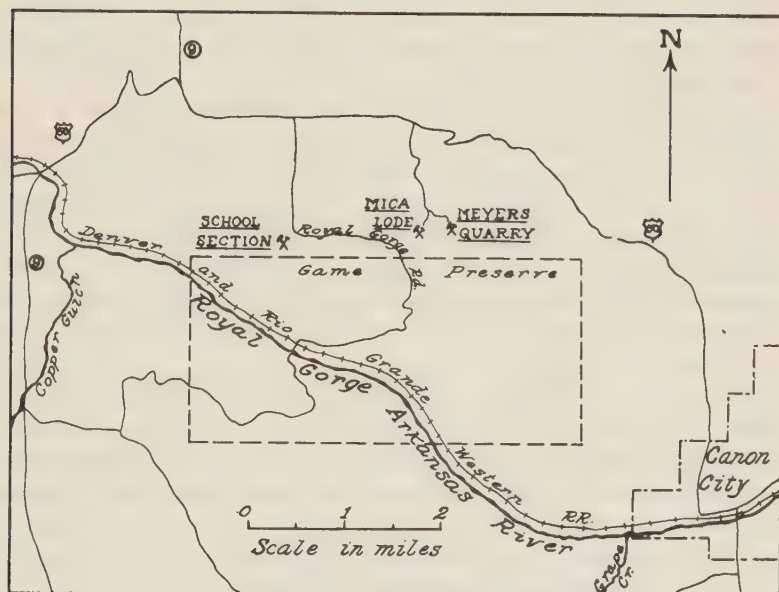


FIG. 1. Index map showing location of Mica Lode and School Section pegmatite deposits, Eight Mile Park, Fremont County, Colorado.

that transect the primary igneous foliation. Two of the three larger pegmatite deposits, the Meyers Quarry body and the Mica Lode, are in schist, and the third, the School Section deposit, occurs in granite. Triplite was found in the Mica Lode and School Section pegmatites. The location of these deposits is shown in Fig. 1. The Mica Lode is in the NE $\frac{1}{4}$, SW. $\frac{1}{4}$, sec. 14, T. 18S., R. 71 W. and the School Section quarry is in the southeast corner of Sec. 16, T. 18 S., R. 71 W.

OCCURRENCE

Triplite in rounded, anhedral masses as much as six inches in length is abundant locally in the School Section pegmatite, but no crystals were

obtained from this deposit. Associated minerals are muscovite, plagioclase, black tourmaline, and beryl. The plagioclase is of two types: pink oligoclase-albite and white cleavelandite. These minerals are concentrated in units of restricted size which underlie flat-lying to horizontal core pods. The core pods, or central zones within the pegmatite, are composed of massive white quartz and crystals of pink microcline as much as six feet across. The zonal structure appears to have formed by direct crystallization from the pegmatite magma. The triplite-bearing units, which are clearly secondary, probably formed by hydrothermal replacement of parts of the cores and parts of the adjoining graphic granite-quartz-muscovite zone.

The geologic relations at the Mica Lode are similar, except that the microcline-rich core is a single large unit that dips steeply northwest in conformity with the general attitude of the pegmatite. The footwall half of the core has been extensively replaced by abundant pink oligoclase-albite and muscovite. Beryl, with which the triplite is associated, is abundant locally in this replacement unit. Triplite pods as much as two feet long were observed in place, and about 10 tons of the material has been hand-cobbed and stockpiled. The crystals studied were obtained from the stockpile.

Some nodules of triplite are corroded and veined by albite and muscovite. At the School Section deposit veins of black tourmaline transect the phosphate. In this respect the material is similar to that found at Chatham, Connecticut. This occurrence has been described by Shannon (1921) who states (p. 445), "Intergrown intimately with the triplite are muscovite and fine-grained deep-blue tourmaline, the last surrounding the triplite in a crust and penetrating it along cracks . . ."

In 1945 Dr. Chalmer J. Roy of Louisiana State University sent to the Mineralogical Laboratory of Harvard University a specimen of triplite from El Paso County, Colorado. According to Dr. Roy the mineral occurs in a pegmatite in the southeast corner of sec. 9, T. 16 S., R. 67 W., where it is associated with quartz, microcline, and muscovite.

OCCURRENCE OF TRIPLITE IN THE UNITED STATES

Data on the occurrences of triplite in the United States are summarized in Table 1.

CRYSTALLOGRAPHY

Four crystals from the Mica Lode pegmatite were examined and measured by means of the contact goniometer. The two large ones, shown in Figs. 2 and 3, proved very satisfactory for contact measurement purposes and yielded moderately reproducible results. The dimensions

TABLE 1. TRIPLITE OCCURRENCES IN THE UNITED STATES

No.	Locality	Reference	Occurrence	Probable Origin
1	Stoneham, Maine	Kunz, 1884	pegmatite	hydrothermal replacement
2	Auburn, Maine	Bastin, 1911	pegmatite	hydrothermal replacement
3	Chatham, Conn.	Shannon, 1921	pegmatite	hydrothermal replacement
4	Branchville, Conn.	Dana, 1892	pegmatite	hydrothermal replacement?
5	School Section, Fremont County, Colo.	Wolfe and Heinrich, 1947	pegmatite	hydrothermal replacement
6	Mica Lode, Fremont County, Colo.	Wolfe and Heinrich, 1947	pegmatite	hydrothermal replacement
7	El Paso County, Colo.	Wolfe and Heinrich, 1947	pegmatite	?
8	7U7 Ranch, Arizona	Hurlbut, 1936	pegmatite	?; described as a segregation
9	Mt. Loma and two nearby localities, Arizona	Hurlbut, 1936	pegmatite	?; described as segregations
10	White Pine Co., Nevada	Hess and Hunt, 1913	high temperature vein	hydrothermal deposition

of these two crystals, measured in the directions of the *a*, *b*, and *c* axes, are 9×9×12 cm. and 9×12×9 cm. The other two crystals, which are less well developed, have maximum dimensions of 9 cm. The quality of the angular readings on the smaller crystals is inferior to that for the larger ones.

Quality of measurements and important measured and calculated values for the four crystals are given in Table 2. No elements have been derived from these measurements, because it is believed that the elements derived from *x*-ray values are more satisfactory. The correlation with the structural cell was accurately determined by cutting an oriented fragment from crystal 1, from which rotation and weissenberg data were obtained.

With the elements derived from an *x*-ray examination of an oriented crystal fragment, the following angle table (Table 3) was prepared. All of the forms listed may be considered certain. Although {112} was observed but once, it was clearly found and in good position.

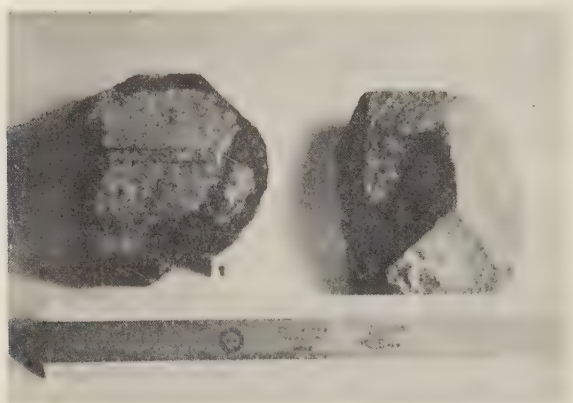


FIG. 2. Photograph of triplite crystals (crystals 1 and 2), from the Mica Lode pegmatite, Eight Mile Park, Fremont County, Colorado.

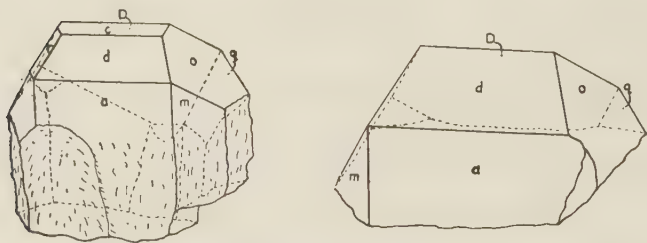


FIG. 3. Crystals of triplite from the Mica Lode pegmatite; left—crystal 1, right—crystal 2.

It is interesting to note that all of the observed forms have indices that conform to the body-centering criteria of $h+k+l=2n$, and the relative importance of the forms is in simple relationship to the spacing of the planes in the centered lattice.

Full development of a prismatic form was not observed. The crystal class is not certain; but, since a plane of symmetry was apparent on crystals 1 and 2, the class must be domatic—*m* or prismatic — $2/m$. The latter is assumed here for lack of evidence to the contrary.

TABLE 2. SUMMARY OF CRYSTALLOGRAPHIC DATA

Crystal 1 (see Fig. 3)						
Faces observed: 001, 100, $\bar{1}00$, 110, $\bar{1}\bar{1}0$, 011, 0 $\bar{1}1$, 101, $\bar{1}01$, 1 $\bar{1}2$, $\bar{2}11$, $\bar{2}\bar{1}1$						
Measured		Calculated				
001 $\wedge$ 011 = 58°		good	55°49'			
100 $\wedge$ 110 = 63°		poor	60°28½'	{001}	cleavage, fair	
100 $\wedge$ 101 = 45°		poor	40°57½'	{100}	cleavage, poor	
101 $\wedge$ $\bar{1}01$ = 79°		good	79°14½'			
101 $\wedge$ $\bar{2}\bar{1}1$ = 47°		fair	46°45'			
011 $\wedge$ 1 $\bar{1}2$ = 29°		poor	27°42'			
Crystal 2 (see Fig. 3)						
Faces observed: 100, $\bar{1}\bar{1}0$, 011, 0 $\bar{1}1$, 101, $\bar{1}01$, $\bar{2}\bar{1}1$						
001 $\wedge$ 011 = 57°		poor	55°49'			
100 $\wedge$ 110 = 63°		good	60°28½'			
100 $\wedge$ 101 = 41½°		good	40°57½'	{001}	cleavage, fair	
101 $\wedge$ $\bar{1}01$ = 82°		fair	79°14½'			
$\bar{1}01$ $\wedge$ $\bar{2}\bar{1}1$ = 49½°		good	46°45'			
Crystal 3 (not figured)						
Faces observed: 001, 100, $\bar{1}00$, 011						
101 $\wedge$ $\bar{1}01$ = 78°		fair	79°14½'			
Crystal 4 (not figured)						
Faces observed: $\bar{1}00$, $\bar{1}01$, $\bar{2}11$						
$\bar{1}00$ $\wedge$ $\bar{1}01$ = 62°		fair	59°48'	{001}	cleavage, good	
$\bar{1}01$ $\wedge$ $\bar{2}11$ = 44½°		poor	46°45'	{010}	cleavage, fair	

TABLE 3. TRIPLITE—ANGLE TABLE

Monoclinic: Prismatic $-2/m$ (?) $a:b:c=1.836:1:1.531$; $\beta=105^\circ53'$; $p_0:q_0:r_0=0.834:1.472:1$ $r_2:p_2:q_2=0.679:0.566:1$; $\mu=74^\circ07'$; p_0' 0.867, q_0' 1.531, x_0' 0.285

Forms:	ϕ	ρ	ϕ_2	$\rho_2=B$	C	A
c 001	90°00'	15°53'	74°07'	90°00'	0°00'	74°07'
a 100	90 00	90 00	0 00	90 00	74 07	0 00
m 110	29 31½	90 00	0 00	29 31½	82 15	60 28½
o 011	10 32½	57 17½	74 07	34 11	55 49	81 08½
d 101	90 00	49 02½	40 57½	90 00	33 09½	40 57½
D $\bar{1}01$	-90 00	30 12	120 12	90 00	46 05	120 12
r 112	43 11	46 23½	54 18	58 08	36 58	60 17½
q $\bar{2}11$	-43 25½	64 37½	145 23½	48 59½	75 59	128 23½

X-RAY DATA

In order to correlate the geometrical crystallography with the x-ray data of Richmond (1940) an oriented section was cut from crystal 1, and rotation, 0-layer line, and 1-layer line pictures were taken about the [010] axis, with copper radiation. The solution of these pictures gave the following results:

	Wolfe-Heinrich	Richmond
Space Group	<i>I</i> 2/ <i>m</i>	<i>P</i> 2 ₁ / <i>a</i>
<i>a</i> ₀	11.90	12.03
<i>b</i> ₀	6.48	6.46
<i>c</i> ₀	9.92	10.03
β	105°53'	105°42'
Measured gravity	3.64	3.84
Calculated gravity	3.64	3.94
Mg:Mn:Fe=	2:3:1	Fe:Mn=1:8

Richmond's original pictures were reexamined, and the space group is definitely body-centered.

OPTICAL PROPERTIES

The optical properties of the Mica Lode triplite are as follows:

Orientation	<i>n</i>	Pleochroism
X $\wedge$ <i>a</i> = 57°	1.643 $\pm$.003	reddish brown
Y = <i>b</i>	1.647	yellow
Z $\wedge$ <i>c</i> = -41°	1.668	reddish brown

(+); 2*V* = 25° (measured); *r* > *v*, moderate

Table 4 compares the optical properties of the Colorado triplites.

TABLE 4. OPTICAL PROPERTIES OF COLORADO TRIPLITES

	School Section	Mica Lode	El Paso County
<i>n</i> X	1.671 $\pm$.003	1.643 $\pm$.003	1.648 $\pm$.003
<i>n</i> Y	1.681	1.647	1.652
<i>n</i> Z	1.686	1.668	1.672
2 <i>V</i>	moderate	40°	55°
Sign	(-)	(+)	(+)

CHEMICAL COMPOSITION

The optical properties of the Mica Lode triplite are comparable to those determined by Hurlbut (1936) for the mineral from the 7U7

Ranch, which contains nearly 12% MgO and has an Mg:Mn:Fe ratio of 2:3:1. The specific gravity for triplite of that composition as calculated with our x -ray data is 3.64. Several gravity determinations, made by means of the Berman balance on pure fragments of the Mica Lode triplite, gave an average value of 3.64. A qualitative test indicated the presence of relatively large amounts of magnesium in this triplite.

Complete analyses of both the Mica Lode and the School Section triplites are to be made in the future. It is certain, however, that triplite can no longer be regarded as a simple series between Fe'' and Mn, as indicated by Otto (1936) and Richmond (1940). In some triplites magnesium enters into the composition in important quantities; and, as Hurlbut (1936, p. 656) noted, the optical and other physical properties clearly reflect this variation.

ALTERATION

Triplite is commonly altered to black manganese oxide which coats the crystals and stains the surrounding minerals. Most of the triplite crystals and pods also are enclosed in a thin shell of altered material which lacks the usual vitreous luster and is also somewhat darker in color. This outer skin has been variously described as "pseudotriplite" or a "substance near heterosite" (Dana, 1892).

The material of the outer shell has an n_z index of refraction considerably lower than that of triplite (in the Mica Lode mineral, 1.600 as compared to 1.668), but x -ray powder photographs of the shell and the fresh triplite core are identical. The shell probably represents the initial stage in the breakdown of the triplite. The isostructural decomposition product may be the result of partial oxidation of the iron and manganese. Under the microscope the outer material is nearly opaque owing to the presence of very abundant minute dark brown inclusions.

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OPTICAL FIGURES OBTAINED WITH THE REFLECTING MICROSCOPE

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ABSTRACT

Optical figures, obtained with a petrographic microscope converted to a reflecting microscope, are described. Four types of optical figures were seen. These are: (1) type I, which resembles a uniaxial cross, but differs in being centered in all orientations; (2) type II, which resembles a centered acute bisectrix interference figure, differing in being centered for all orientations, although the size of the optic angle shows a relation to orientation; (3) type III, which resembles a centered acute bisectrix interference figure fixed in the 45° position, and which is the same for all orientations; and (4) type IV, which is like type III, except that the isogyres move from a minimum to a maximum optic angle on rotation of the stage. Nothing is known concerning the relation of type IV to different orientations. Type I is subdivided into four subtypes, three of which are based on the width of the isogyres, the fourth subtype differing also in having color bands. Type II is subdivided into two subtypes, subtype IIa being seen only on natural surfaces and subtype IIb only on polished surfaces.

A large part of this investigation has been limited to natural surfaces, because of modifying effects produced by polishing. Other than this effect, the nature of the surface examined governs only the quality of the figure. The test plates, used in petrography, have no effect on the figures. The optical figures from a few minerals show color phenomena and color patterns, some of which phenomena are likened to "dispersion." Rotation of the polarizer affects differently the figures from different minerals. The best figures are ob-

tained from the opaque minerals of metallic luster. Nonmetallic minerals of dark color also yield optical figures, but light colored, nonopaque minerals yield no figures.

A tabular summary of the results obtained from the minerals examined is added at the end of this report.

INTRODUCTION

Optical figures on polished sections of opaque minerals were first noticed by the author when he was studying mineralography, under Dr. Joseph Murdoch of the department of geology at the University of California at Los Angeles. While studying the anisotropism of covellite under a 4 mm. objective on a petrographic microscope modified to a reflecting microscope, the author inserted the Bertrand ocular for the sake of curiosity. A figure, analogous to a centered acute biaxial interference figure was seen. Superficial examination of other minerals showed similar figures. A study of these figures was undertaken as a research problem in the department of geology at the University of California at Los Angeles, in the Spring of 1946.

The original object of this investigation was to determine whether the figures obtained from opaque minerals could be used in the determination of minerals in polished section. This question has not been completely answered, but, because of the apparent complexity, the indication is that these figures will be of only limited use in this connection. It was necessary in attempting to answer this question to determine the number of types of figures, their relations to crystallography, and to compare them with true interference figures obtained from the nonopaque minerals with transmitted polarized light.

The purpose of this report is threefold. It is intended (1) to show the general nature of the optical figures seen in this investigation, (2) to demonstrate the desirability of further study, and (3) to form a basis for further investigations of these phenomena. No attempt is made to explain the observed phenomena, this report being solely a consideration of the observations made; however, some inferences are made with respect to expected behaviors of the figures in a few cases.

No previous literature mentioning optical figures from opaque surfaces was found, although no exhaustive search of the literature was attempted. That these figures are not entirely unknown is apparent from the fact that Dr. R. W. Webb informed the author that he and others had noticed the figures and recognized them to be some type of optical figure, but had not investigated them.

The author gratefully acknowledges the guiding influences and many valuable discussions offered by Dr. Joseph Murdoch and Dr. Cordell Durrell, of the department of geology at the University of California at Los Angeles. Thanks are due Dr. Durrell for critically reading

this manuscript. The author also wishes to express his appreciation for the interest shown in this work by Dr. George Tunell, of the Geophysical Laboratory and for his encouragement in presenting this report for publication. This investigation was carried out under the supervision of Dr. Murdoch.

TECHNIQUES AND EQUIPMENT

The microscope employed was a Spencer petrographic microscope, model 42A. This was converted into a reflecting microscope by installing a Bausch & Lomb vertical illuminator. As shown in Fig. 2 the illuminator is of the transparent disk rather than the prism type. A 4 mm. short-mounted objective was used. The lamp employed was a Bausch & Lomb projection type, water cooled incandescent lamp, using a ribbon filament,

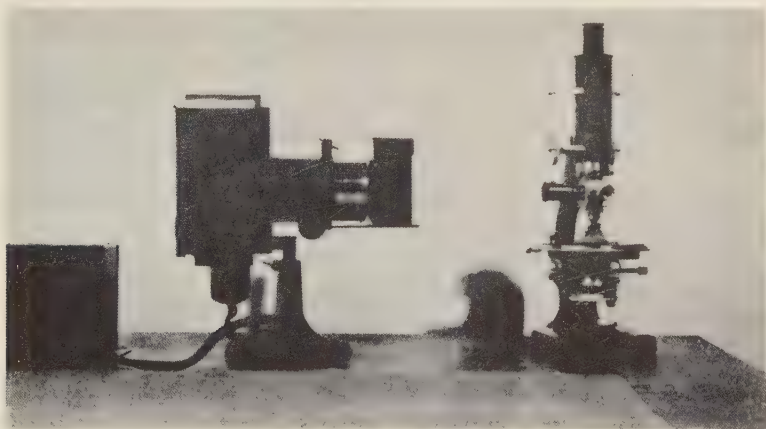


FIG. 1. The equipment as used in this investigation. A crystal of rutile, with (100) in reflecting position, mounted in plasticene, is seen on the stage of the microscope.

projection type bulb; in two cases a coil filament bulb was used. The mirror used was an optically-parallel-ground piece of glass. The equipment is shown in Fig. 1.

The importance of a strong source of illumination can hardly be over-emphasized, for otherwise the figures are visible only on a few minerals of very strong anisotropism, such as covellite. Strongest illumination is obtained by setting the polarizer so as to bring the light in from the side of the microscope as shown in Fig. 1. In this position the vibration direction of the polarized light is set parallel to the mirror. This is diagrammatically shown in Fig. 2, in comparison with what is apparently the usual setup. By this means the amount of light reflected from the plane

glass surface is much greater in comparison to that passing through, than is the case with the usual setup.

The lens system in the equipment employed was not very satisfactory, for reflections from surfaces in the system produced blind spots in the field. This was partly obviated by increasing the distance between the light source and the polarizer, but beyond a certain distance this causes a decrease in the area of light on the field of view.

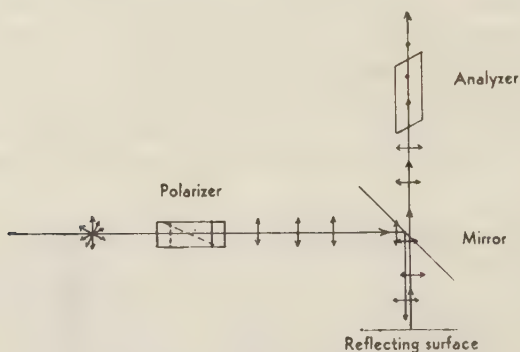


FIG. 2a. The relation between the vibration direction and the mirror in the usual setup.

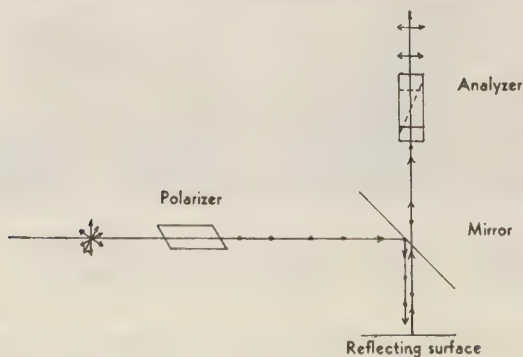


FIG. 2b. The relation between the vibration direction and the mirror in the setup used in this investigation.

The techniques and mounts used with polished surfaces are the same as described by Murdoch (*Econ. Geol.*, **33**, 542, 1938). Crystals and cleavage fragments are mounted in plasticene, in such a way that the base of the mount and the surface being examined are optically parallel. This is often difficult to accomplish because of irregularities in the surface or

because of the small area of a surface in relation to the rest of the grain. There is a very definite limit of about 3 mm. diameter below which the fragment or crystal could not be mounted by this method.

In examining a mineral surface, the mounted specimen is first placed on the stage of the microscope, the 4 mm. objective is brought into focus, and the Bertrand ocular is inserted. It is necessary, with the equipment employed, at this stage to adjust the position of the lamp in order to eliminate or decrease the size of the blind spots in the field due to the optical system. The type of figure, the effects due to the nature of the surface, rotation of the stage, effects due to test plates, and other phenomena, such as "dispersion" are then noted. The optic angle, where there is one, is estimated on the basis of the field diameter being equivalent to a 60° optic axial angle. As many orientations as possible from the available material for each mineral are investigated.

NATURE OF THE SURFACES EXAMINED

Two general types of surfaces were examined: polished and natural. The polished surfaces are generally highly polished and mirror-like; the only marring feature was the presence of small scratches in some specimens.

The crystal surfaces examined showed the usual imperfections, including symmetry pits and ridges, small cracks and steps. Small cracks on minerals which are not truly opaque are very disturbing, as they give intense internal reflections in certain positions between crossed nicols, which seriously interfered with the quality of the figures. Cleavage surfaces are generally not satisfactory, as they are stepped and show other breakage phenomena. It was usually difficult to find a single area on cleavage surfaces large enough to yield a figure. It is necessary to have an area at least about one-fourth the area of the field when viewed without the Bertrand lens.

DEFINITIONS OF TERMS

The quadrants in the field of view, the optic angle, the crossed and the 45° positions, the vibration directions of the polarizer and analyzer, and the diameter of the field of view of the microscope used are diagrammatically shown in Fig. 3. The positive and negative directions of measurement of angles on rotation of the stage are also shown.

The use of the terms optic angle, optical figure, and dispersion may not be merited as the optics involved in these figures are not known, but the terms are used here because of the close similarity of these optical figures to interference figures obtained with transmitted light.

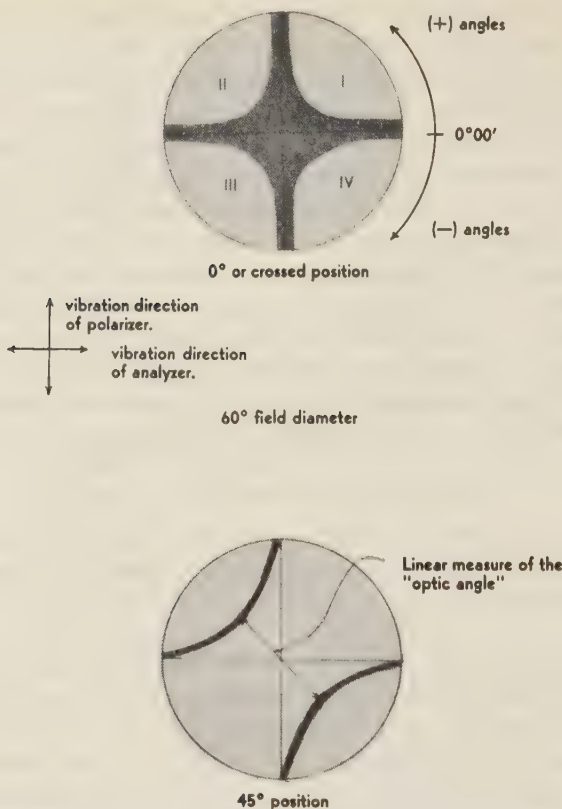


FIG. 3. Diagrammatic definition of some terms.

DESCRIPTIONS OF TYPES OF OPTICAL FIGURES

TYPE I. Type I in general form is like the centered uniaxial optic axis interference figure, but it is centered in all orientations.^f Type I is subdivided into four subtypes (Fig. 4). Three of the subdivisions are based on the width of the isogyres; they grade into one another. The fourth subtype differs from the others in having color bands. The width of the isogyres is perhaps related to optical constants in much the same way that the width of the isogyres in the uniaxial interference figure is related to birefringence. It appears that one mineral species may show more than one of these subtypes.

Subtype Ia has thin isogyres. It has been seen only on polished surfaces of minerals. This figure has been found on a fine-grained aggregate of breithauptite and niccolite, and on chalcopyrite, cuprite, and pyrite.

Subtype Ib has isogyres of medium thickness, about like that of the

centered obtuse bisectrix figure in the crossed position. It was the commonest subtype noted and was found on both natural and polished surfaces. It is perhaps the most characteristic figure obtained from dark-colored nonmetallic minerals, and has been found on crystal faces of andradite, augite, diopside, grossularite, magnetite, and sphalerite. It was also found on polished surfaces of boulangerite, bournonite, chalcocite, cosalite, gratonite, magnetite, and polybasite (?).

Subtype Ic has very broad isogyres, which closely resemble the optic normal interference figure. It was found on crystal faces of boléite and sphalerite, and on polished surfaces of pyrrhotite and sphalerite.

Subtype Id is like subtype Ia, with respect to the width of the isogyres, but it differs in being the only subtype of type I showing any color pattern. This pattern consists of color bands at 0° and 180° which are convex toward the center of the field. The colors range from second order blue green to second order red in Newton's series of colors (see Fig. 4). This subtype was seen only on one grain of pyrite in a polished section from the Kelly Gold and Silver Mine, Randsburg, California.

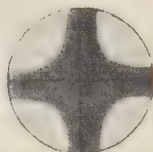
TYPE II. The general form of type II is like a centered acute bisectrix interference figure. It shows a 90° phase relation, being in the crossed position four times and in the 45° position four times through 360° of rotation. In the crossed position the figure resembles subtype Ib.

This type is subdivided into two subtypes (see Fig. 5) based on the different behaviors on polished surfaces versus natural surfaces. On polished surfaces the analogy of the optical figure to the centered acute bisectrix interference figure is not so close, for the optic angle in adjacent 45° positions is, in most cases, not the same. The figures of subtype IIa are from natural surfaces and those of subtype IIb from polished surfaces.

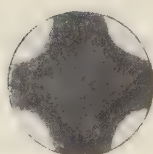
Subtype IIa shows a general relation of the size of the optic angle to orientation. The optic angle is largest in the prism zone and becomes smaller as the section approaches perpendicularity to the base and on the base the optic angle is 0° . There are exceptions to this general rule as is shown by epidote (see appendix). The exceptions may perhaps be explained in the same way as is the occurrence of the acute bisectrix interference figure being in different positions, with respect to the crystallographic axes, on different biaxial minerals. On hexagonal and tetragonal minerals the optic angle is the same for all faces in the prism zone, whereas in minerals of lower symmetry, such as wolframite, the optic angle is at a maximum on one pinacoid in the prism zone and at a minimum value in a direction at right angles (?) to it. This is perhaps one of the most valuable observations from this study, for it gives a means of determining whether a given opaque mineral is tetragonal or hexagonal on the one hand, or orthorhombic, monoclinic or triclinic on the other,



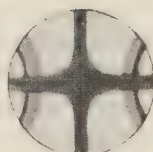
Subtype 1a.



Subtype 1b.



Subtype 1c.

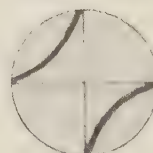


Subtype 1d.

FIG. 4. Type I.



0°



45°

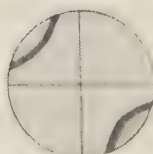
Type IIa



(-) 45°



0°



(+) 45°

Type IIb.

FIG. 5. Type II.

as, with one other possible exception, the type of figure does not seem to indicate the crystal symmetry. Whether this observation will hold for polished sections is not known. The crystals showing subtype IIa and their estimated optic angles are azurite (10°), brookite (5°), cassiterite (7°), chalcopyrite (10°), covellite (65°), epidote (5°), hematite (45°), ilvaite (5°), marcasite (10°), rutile (20°), stephanite (?), and wolframite (15°). Many of these angles are probably not the maximum values, for only one orientation or an insufficient number of orientations were available in examination of many of the minerals. It is tentatively concluded on the basis of these few observations that those minerals with the most marked anisotropism, such as covellite and hematite have the largest optic angles.

Subtype IIb, seen only on polished sections, appears to be related to subtype IIa by some modifying effect induced by polishing. The main difference between subtypes IIa and IIb is the marked asymmetry of the optic angle in subtype IIb, wherein it may be greater in quadrants I and III, than in quadrants II and IV, or the reverse. The asymmetry of the optic angle is not seen in all sections, as shown by enargite. It is not known whether the asymmetry of the optic angle bears any definite relation to the orientation of the surface. This subtype apparently shows the same relation of the size of the optic angle to orientation as does subtype IIa.

Figures of subtype IIb were found on polished surfaces of arsenopyrite, covellite, enargite, löllingite, miargyrite, specularite, and stephanite. (See appendix for the values of the optic angles.)

TYPE III. Type III has been noted only on natural surfaces, and, with two exceptions, only on isometric minerals. It is like the centered acute bisectrix interference figure in the 45° position, but differs in that the isogyres do not move from this position throughout 360° of rotation. The isogyres were found only in quadrants I and III in all cases examined (see Fig. 6). The figure is the same for all orientations.

Type III appears to be fundamentally related to isometric symmetry, although limited to metallic opaque or near-opaque minerals, regardless of the two exceptions. It has been found on a specimen labeled glaucodot, and on molybdenite. The glaucodot may, however, be replaced by pyrite, the figure being very much like that of pyrite. The figure from molybdenite is not so easily explained.

Type III is seen on cuprite (15°), franklinite (20°), galena (15°), pyrite (25°), tetrahedrite (5°), and the two non-isometric minerals glaucodot (20°) and on a (0001) cleavage surface of molybdenite. No other orientations of molybdenite were available.

Although magnetite shows an optical figure of type I, it may conceiv-

ably be type III in which the optic angle is 0° , for it is the only metallic isometric mineral examined that did not show a type III figure on a natural surface.

TYPE IV. Type IV has been noted only on natural surfaces. It consists of two isogyres in quadrants I and III that are convex toward the center of the field; the isogyres move from a minimum to a maximum optic angle on rotation of the stage (see Fig. 7). Presumably this minimum angle may be as low as 0° , although it was not noticed on the minerals showing this type: The most startling fact which was observed in connection with this type is that, totally unlike biaxial interference figures and type II figures, the figure returns to the same position only every



FIG. 6. Type III.



FIG. 7. Type IV.

180° of rotation. On each of the minerals examined, only one orientation was available so that no information concerning a relation to orientation could be ascertained. This type was seen only on arsenopyrite and stibnite in which the minimum and maximum angles are respectively 5° and 25° , and 15° and 50° .

At this point it should be noted that type II*b*, at least in the case of arsenopyrite, is derived by the effects of polishing not only from subtype II*a*, but also from type IV.

In a speculative mood, it might be supposed that type III and type IV show a fundamental relation to each other, similar to that which types I and II show. That is, type IV may in certain orientations take on the form of type III, as type II does with respect to type I. If this were true, it might well explain the two apparent exceptions to the statement that type III is characteristic only of the metallic isometric minerals.

EFFECTS DUE TO TEST PLATES

The mica plate and the quartz wedge produce only a uniform color over the field, dependent on their thickness and double refraction. The gypsum plate produces a constant color pattern for all minerals, except diopside and epidote. The color pattern (Fig. 8) remains the same throughout 360° of rotation. For diopside and epidote the gypsum plate produces only a sensitive tint uniform over the whole field.

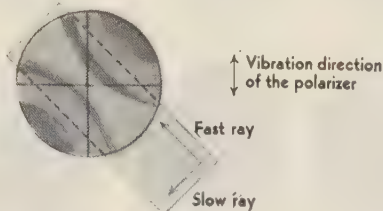


FIG. 8. Effect of the gypsum test plate.

"DISPERSION" AND COLOR PHENOMENA

The term "dispersion" is used here, as that is the nearest thing known to the author with which the phenomena under consideration may be compared. Three general phenomena are considered here.

The most interesting and striking of these phenomena are the color patterns observed on a few minerals. Rather than attempting to describe these color patterns, they are shown diagrammatically in Figs. 9, 10, 11, and 12; it was found impossible to reproduce accurately the actual colors. The color patterns show a definite, regular relation to the isogyres and to rotation of the stage, Hematite appears to have a different color pattern on different faces, whereas the color pattern on covellite is the same for all orientations. Color patterns are apparently not affected by polishing. The color bands on pyrite, showing the subtype *Id* figure, may perhaps logically be included in this group of color pattern phenomena. Type III figures show no color patterns on any of the minerals examined. It is of interest to note that the salmon pink in the color pattern of covellite is precisely the same shade seen as one of the rotation colors between crossed nicols.

On certain minerals of types II, III IV, a color phenomenon was noticed that resembles the dispersion of the optic angle in biaxial interference figures. The colors involved are a dirty red-brown and blue-gray. The red band is very narrow and on the concave side of the isogyres, whereas the blue-gray fills the area between the isogyres (Fig. 13).

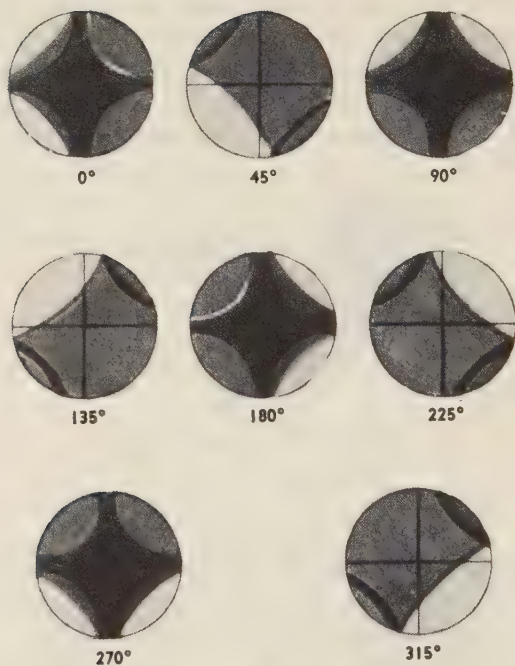


FIG. 9



FIG. 10

FIG. 9. Color pattern on covellite; "optic angle" as on an assumed $(h0\bar{h}l)$ face.

FIG. 10. Color pattern on enargite; "optic angle" as on an $(hk0)$ face.

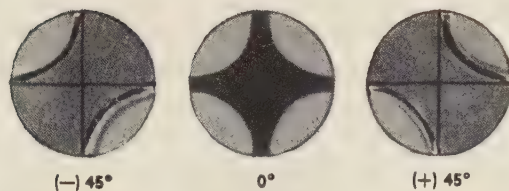
FIG. 11a. Color pattern on hematite on $r(10\bar{1}1)$.FIG. 11b. Color pattern on hematite on a steep $(h0\bar{1}l)$ face.

FIG. 12. Color pattern on stibnite.



FIG. 13. "Dispersion" of the optic angle.

In all types the isogyres are subject to variations in intensity of darkness only in certain positions during rotation of the stage. This effect consists of an extreme lightening of the color of the isogyres at this point to a very light gray; not uncommonly this is limited to the center portion of the isogyres (Fig. 14). For the sake of brevity, this effect is termed "dispersion of the isogyres" in the tables in the appendix. This phenomenon appears to be a property of the mineral. Perhaps related to this is the



FIG. 14a. "Dispersion of the isogyres" in the crossed position.

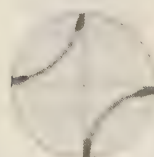


FIG. 14b. "Dispersion of the isogyres" in the 45° position.

phenomenon in type IV, where, at the minimum angle position, the isogyres may be a very dark shade of a certain color, and at the maximum angle position they may be a very dark shade of a different color. This is well shown by stibnite, whose isogyres are broad and very dark red brown at the minimum angle position and thin and very dark blue at the maximum angle position (see Fig. 12).

RELATIONS TO LUSTER AND DEGREE OF OPACITY

Minerals of metallic luster and a high degree of opacity give the best and most varied figures, and this study is primarily based on such minerals. The smoothest surfaces give the clearest figure so that the best figures are obtained from polished surfaces, but because of the effects of polishing most of the investigation was limited of necessity to natural surfaces.

With respect to the truly nonmetallic minerals, the best and most varied figures are obtained from those minerals with the highest luster and the darkest color. It is interesting to note that with few exceptions dark-colored minerals, with vitreous or slightly higher luster, seem to give subtype Ib or Ic figures. The exceptions gave subtype IIa figures in which

the optic angle was 10° or less. Light colored minerals of any degree of luster give no figure. This is perhaps due to a high degree of internal reflection, which gives only a hazy field of a paler shade of the same color as the mineral.

EFFECTS DUE TO ROTATION OF THE POLARIZER

Rotation of the polarizer, with the stage at a set position, causes either of two effects depending on the mineral. On some minerals the only effect is to cause the intensity of the color of the isogyres to fade with continued rotation, and eventually to cause the isogyres to disappear. The positions of the isogyres during rotation of the polarizer do not change.

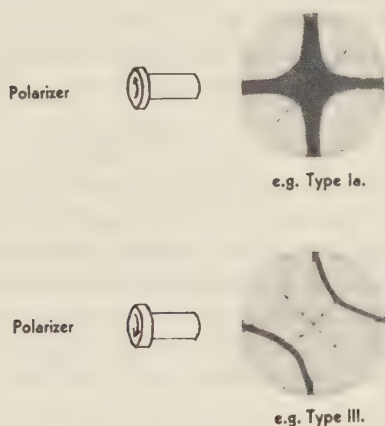


FIG. 15. Directions of movements of isogyres on rotation of the polarizer.

On other minerals the effect is to cause what basically can be assumed to be a change of type in the cases of types I and III. During rotation of the polarizer, the isogyres move in directions controlled by the direction of rotation of the polarizer as shown in Fig. 15. On type III, the isogyres may enter quadrants II and IV, where they were not seen to occur otherwise. Continued rotation of the polarizer not only causes further movement of the isogyres, but causes the isogyres to disappear in the same manner as in the first effect, usually before they have passed out of the field. These effects were investigated only on a few of the minerals examined.

During the early part of the investigation the minerals were examined with the nicols set at the crossed position by judgement of the eye, which is not very accurate, but no other way was at hand at the time. Once

the polarizer had been set in position, it was not disturbed. Later the effect of rotation of the polarizer was investigated at Dr. Murdoch's suggestion. It was found then that magnetite gave a figure of type I while at the most accurate position obtained by judgement of eye. The figure changed to type III on rotation of the polarizer. For the rest of the investigation, magnetite in the position where it gives a type I figure was used as a means of adjusting the nicols in the crossed position. Thus magnetite is the standard for determining the type and optic angle of most of the minerals.

It has been noted that subtype II*b* may be derived not only from subtype II, but also from type IV. The known effects of rotation of the polarizer suggest the possibility that type IV is derived from type II*a* by rotation of the polarizer. However, as a standard setting of the polarizer is chosen, which is believed as near at 90° to the analyzer as possible with the present equipment, this is considered to be a valid type.

EFFECTS DUE TO POLISHING

It was recognized early that polishing causes a change in the figures obtained from various minerals. At the beginning of this study, the investigation was limited to polished surfaces, until an unusually excellent crystal of rutile was examined out of curiosity. It was noted on this crystal that the type II figure was not asymmetric with respect to the optic angle, whereas all the type II figures which had been seen previously were. This was investigated further, and found to be invariably true; therefore all subsequent work was limited to natural surfaces, it being felt that this was necessary before any attempt at a systematic study of the polished surface figures could be started. Much more work in this direction is yet to be done before such a study can be attempted.

With respect to the change of type, which polishing seems to produce, all type III figures are changed to type I, and types II*a* and IV figures to subtype II*b*. The asymmetry of the optic angle seen in subtype II*b* is apparently caused by polishing as it has never been observed on natural surfaces.

The most striking effect produced by polishing consists of an aggregate effect, wherein a very fine-grained aggregate of one or more minerals, any individual of which is too small to give a figure, yields a figure of either type I or subtype II*b*.

CONCLUSIONS

1. Opaque minerals yield figures by reflected polarized light that are analogous to interference figures obtained by transmitted polarized light in nonopaque minerals.

2. The figures obtained may be classified into four principle types and into six subtypes.

3. The four subtypes of type I are based on the width of the isogyres and on the presence of color bands. The two subtypes of type II are based on the difference in configuration on natural surfaces and polished surfaces.

4. Figures obtained from polished surfaces are different from those obtained from crystal faces of the same mineral, and this is believed due to the mechanical effects of polishing.

5. Other than the effects due to polishing, the nature of the surfaces governs only the quality of the figure.

6. The test plates produce no changes that might be of value in identifying the mineral.

7. Certain minerals show color patterns having definite relations to the figure and to rotation of the stage, which are totally unlike the interference colors obtained in interference figures from nonopaque minerals by transmitted polarized light.

8. Some minerals show a color pattern like the dispersion of the optic angle in interference figures from nonopaque minerals.

9. On some figures the isogyres are subject to variations in intensity of darkness only in certain positions during rotation of the stage.

10. Minerals of a high degree of opacity and luster with smooth faces give the best figures. Some nonopaque minerals of dark color yield figures.

11. Rotation of the polarizer affects the figures from different minerals differently.

In conclusion, the author believes that this subject offers a very fruitful line of investigation.

APPENDIX

TABLE 1. FIGURES YIELDED BY NATURAL SURFACES
OF THE MINERALS EXAMINED

Note: This table is arranged with respect to types of figures and according to the crystal symmetry within the types.

TYPE I			
Mineral	Crystal System	Subtype	Remarks
Andradite	Isometric	Ia	On (110)
Grossularite	Isometric	Ib	On (110)
Magnetite	Isometric	Ib	On faces (100) and (111) and on parting (111). "Change of type" on rotation of the polarizer. The standard for setting the polarizer at 90° to the analyzer.
Sphalerite	Isometric	Ib	On rounded (111) face.
Sphalerite	Isometric	Ic	On (110) cleavage.
Boléite	Tetragonal	Ic	On (001)?
Augite	Monoclinic	Ib	On several faces: (110), (100), (010), (111), and (111).
Diopside	Monoclinic	Ib	No effect with the gypsum plate. Several orientations.
TYPE IIa			
Mineral	Crystal System	Optic Angle	Remarks
Cassiterite	Tetragonal	(110) 7° (hkl) 1-2°	Internal-reflecting cracks cause much interference.
Chalcopyrite	Tetragonal	(hkl) 10°	Hazy figure. Suggestion of a color pattern, which could not be resolved.
Rutile	Tetragonal	(hkl) zone 20° flat (hkl) 0° (111) 5°	"Dispersion of the isogyres" in the crossed position.
Covellite	Hexagonal	(0001) cl. 5° (1010) cl. 65°+	Color pattern (see Fig. 9).
Hematite	Hexagonal	(0001) 0° (1011) 10° steep (h0hl) 45°	No color pattern. Color pattern (Fig. 11a). Color pattern (Fig. 11b)
Brookite	Orthorhombic	(122) 5° (110) 0°	"Dispersion of the isogyres" in the crossed position and in the 45° position in quadrants II and IV.
Ilvaite	Orthorhombic	(110) 5°	"Change of type" with rotation of polarizer.
Marcasite	Orthorhombic	(hkl) 10°	
Azurite	Monoclinic	(101) 10° (001) 5°	Strong blue tint on the isogyres.
Epidote	Monoclinic	(101) 5° (011), (010) 0°	No effect on the gypsum plate.
Wolframite	Monoclinic	(110) 5° (010) cl. 15°	"Dispersion of the isogyres" in the crossed position.

TYPE III. (This type is seen only on natural surfaces.)

Mineral	Crystal System	Optic Angle	Remarks
Cuprite	Isometric	(111) 15°	"Dispersion of the isogyres."
Franklinite	Isometric	(111) 20°	Isogyres wide. "Dispersion" of the optic angle strong.
Galena	Isometric	(111) 15° (001) 15°	
Pyrite	Isometric	(001) 25° (110) 25° (111) 25°	No "change of type" on rotation of the polarizer.
Tetrahedrite	Isometric	(111) 5°	"Change of type" on rotation of the polarizer.
Molybdenite	Hexagonal	(0001) 17°	"Dispersion" of the optic angle strong.
Glaucodot	Orthorhombic?	(110) 20°	Replaced by pyrite? "Dispersion of the isogyres." No "change of type" on rotation of the polarizer.

TYPE IV

Mineral	Crystal System		Optic Angle		Remarks
			Min.	Max.	
Stibnite	Orthorhombic	(010) cl.	15°	50°	Color pattern (Fig. 12); the blue color disappears rather sharply at 45°. At 45° the optic angle is 40°. "Change in type" with rotation of the polarizer.
Arsenopyrite	Triclinic?	(110) (1 $\bar{1}$ 0)	5° 5°	25° 25°	"Dispersion of the isogyres" in the 90° position.

The following light-colored, nonopaque minerals, which yielded no figures, were examined: calcite (suggestion of a uniaxial interference figure on a cleavage fragment), pyromorphite, quartz, siderite, titanite, vanadinite (suggestion of a uniaxial figure on (001)), wulfenite, and zircon.

TABLE 2. FIGURES YIELDED BY POLISHED SURFACES
OF THE MINERALS EXAMINED

TYPE I			
Mineral	Crystal System	Sub-type	Remarks
Cuprite	Isometric	Ia	Red isogyres because of strong red internal reflections?
Magnetite	Isometric	Ib	
Pyrite	Isometric	Ia	
Pyrite	Isometric	Id	A coil filament lamp was used in this case.
Sphalerite	Isometric	Ic	
Chalcopyrite	Tetragonal	Ia	
Breithauptite and niccolite	Hexagonal	Ia	A fine granular aggregate. This is an aggregate effect.
Gratonite	Hexagonal	Ib	On (0001), (10 $\bar{1}$ 0), and (11 $\bar{2}$ 0).
Pyrrhotite	Hexagonal	Ic	A coil filament lamp was used in this case.
Boulangerite	Orthorhombic	Ib?	Grains too small.
Bournonite?	Orthorhombic	Ib	
Chalcocite	Orthorhombic	Ib	A fine-grained aggregate of granular chalcocite and feathery covellite. An aggregate effect?
Cosalite	Orthorhombic	Ib	
Polybasite?	Monoclinic	Ib	
TYPE IIb			
Mineral	Crystal System	Optic Angle	Remarks
Covellite	Hexagonal	30° and 40° 40° and 65° 40° and 45°	Several random sections. Color pattern (see Fig. 9).
Hematite var. specularite	Hexagonal	20° symmetrical	A multitude of plates of varying orientations; an aggregate effect.
Enargite	Orthorhombic	5° and 25° 20° symmetrical 5° symmetrical	Several sections. Color pattern (see Fig. 10).
Löllingite	Orthorhombic?	10° and 20°	"Dispersion" of the optic angle.
Stephanite	Orthorhombic	0° symmetrical 5° symmetrical	Reddish isogyres; strong red internal reflections.
Miargyrite	Monoclinic	5° and 10° 0° symmetrical	Random sections. "Dispersion of the isogyres" on the grain where the optic angle is 0°.
Arsenopyrite	Triclinic?	5° and 10°	Wide isogyres.

PUMICE FROM HAYLMORE, BRIDGE RIVER, BRITISH COLUMBIA*

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ABSTRACT

Dacite pumice of Recent age near Haylmore's placer mine, Bridge River district, British Columbia, is highly vesicular and also shows pronounced fluidal structure. The pumice is vitrophyric; the volcanic glass of the groundmass has a refractive index of 1.497. The principal phenocrysts are plagioclase feldspars ($\text{Ab}_{70}\text{An}_{30}$) characterized by an abundance of glass inclusions. Possible modes of origin of the inclusions are discussed. A chemical analysis of the pumice and the calculated norm are given.

Pumice forms a widespread blanket over much of the Bridge River area of western British Columbia, and its occurrence has been briefly noted in reports of the Canadian Geological Survey. Early in 1946

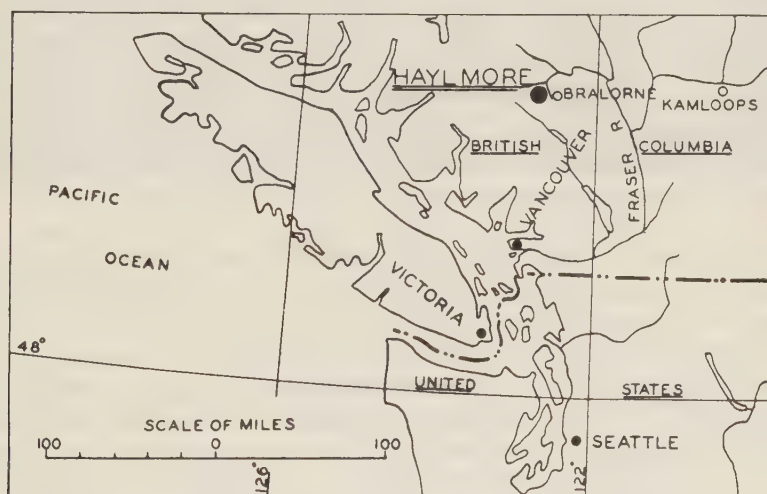


FIG. 1. Index map of British Columbia showing location of Haylmore.

Bralorne mine constructed a new wood road across the Hurley River near its confluence with the Bridge River, exposing an excellent cross-section of the pumice on the west side of the Hurley River, adjacent to the placer workings of William Haylmore (Fig. 1). The pumice rests on stream boulders and silty gravels of Recent age, and grades upward into the soil. The Bridge River pumice bed is remarkably uniform, and

* Presented December 27, 1946, at the annual meeting of the Mineralogical Society of America held in Chicago, Ill.

the Haylmore exposure seems to be a typical section. It is unique, however, in that part of the section shows stratified silty sand overlying the pumice, a sequence not heretofore recorded.

Bateman¹ in 1912 suggested that the source of the pumice was some volcanic vent between the headwaters of the Bridge and Lillooet Rivers, but was unable to locate a vent. McCann² in 1922, and Cairnes³ in 1937, quoted this suggestion, but no vents appear to have been discovered. Lay,⁴ who devoted seventeen years, from 1925 to 1941, largely to a study of the geology of the Fraser River area to the east of the Bridge River, noted the rarity of siliceous lavas and the difficulty of determining the source of acid tuff beds in that region. Recently Cummings has made some investigations of pumice along the headwaters of the Lillooet River, and found that Meagher Mountain, which is situated immediately west of the junction of the South Fork and the main Lillooet River, is at least a local source of pumice. He says, however, "It is debatable whether Meagher Mountain was the source of the Bridge River pumice or merely one of a number of local sources."⁵ Thus the exact source of the Haylmore pumice remains undetermined, but it does appear to have come from the west, and to have been borne along by the prevailing westerly winds.

The Haylmore pumice bed averages two feet in thickness, and consists of a mixture of small and large particles, mostly of the size of sand and gravel, but ranging from fine dust to lumps two inches in diameter. The deposit seems to be the "granular pumice" type of Moore,⁶ and the "pumice fall" of Kôzu as used by Williams.⁷ The pumice is grayish-white with phenocrysts of dull white plagioclase and glistening black hornblende embedded in a highly cellular groundmass of volcanic glass.

Microscopically the rock shows fluidal structure with a subordinate development of radial arrangement of glass out from the blowholes (Fig. 2). Approximately 95 per cent of the rock is groundmass, glassy

¹ Bateman, A. M., Lillooet Map-area, British Columbia: *Canadian Geol. Survey, Summary Rept.*, 193 (1912).

² McCann, W. S., Geology and mineral deposits of the Bridge River Map-area, British Columbia: *Canadian Geol. Survey, Mem.* 130, 43 (1922).

³ Cairnes, C. E., Geology and mineral deposits of Bridge River Mining Camp, British Columbia: *Canadian Geol. Survey, Mem.* 213, 40 (1937).

⁴ Lay, Douglas, Fraser River Tertiary drainage-history in relation to placer-gold deposits: *Brit. Col. Dept. of Mines, Bull. No.* 3, 12 (1940).

⁵ Cummings, J. M., Personal communication, October 29, 1946.

⁶ Moore, B. N., Nonmetallic Mineral Resources of Eastern Oregon: *U. S. Geol. Survey, Bull.* 875, 149 (1937).

⁷ Williams, Howel, The geology of Crater Lake National Park, Oregon: *Carnegie Inst., Washington, Pub.* 540, 68 (1942).

with a few irregular spherulites and tiny microlites. Plagioclase phenocrysts make up 4 per cent of the rock, and mafic minerals account for about one per cent. Orthoclase was not found, nor was quartz. The plagioclase is oligoclase-andesine, with an average composition of $Ab_{70}An_{30}$; $\alpha=1.546$, $\beta=1.550$, $\gamma=1.553$. The plagioclase ranges in size from microlites to crystals 2.50 mm. in length, but averages about 1 mm. Some of the phenocrysts show normal zoning, occasionally as a thin skin of more albitic plagioclase surrounding the crystal. Although hornblende was more conspicuous macroscopically, pale green augite is present in

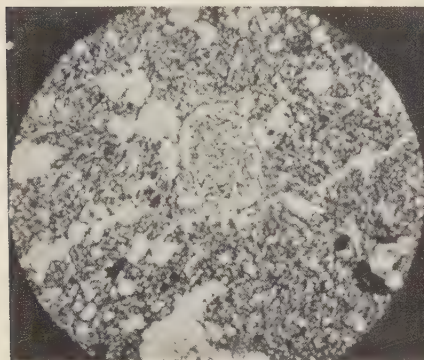


FIG. 2

FIG. 2. Thin section showing the typical rock texture, plagioclase phenocryst in center. Plane light. $\times 15$

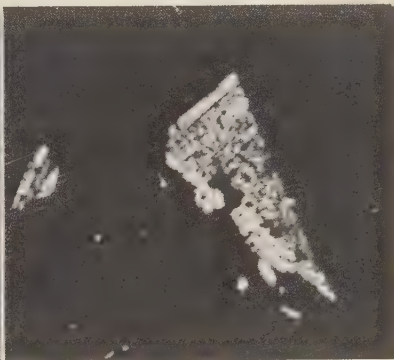


FIG. 3

FIG. 3. Thin section with corroded plagioclase phenocryst, zoned. Crossed nicols. $\times 15$

approximately equal quantity, and a few corroded flakes of dark brown biotite are also found. Hornblende, showing green to brown pleochroism, has the indices $\alpha=1.655$, $\beta=1.675$, $\gamma=1.680$. Augite has $\alpha=1.685$, $\beta=1.689$, $\gamma=1.708$. The minerals present suggest that the rock is a dacite pumice. It appears to be petrographically similar to other such pumices found along the volcanic belt of the Pacific Coast.

The most conspicuous feature of the rock, microscopically, is the abundance of glassy inclusions in the feldspars. Most of the phenocrysts have a worm-eaten appearance, and are cracked and corroded and embayed by the volcanic glass (Fig. 3). Similar glassy inclusions in feldspars have been described many times in other Western volcanic rocks, but their genesis is not readily explained. The inclusions seem to be characteristic of rocks with a glassy groundmass, and especially pyroclastics.⁸

⁸ Coombs, H. A., Personal communication, Nov. 5, 1946. Williams, Howel, Personal communication, Jan. 20, 1947.

Buerger⁹ has suggested that the glassy inclusions may be an expression of lineage structure in the original crystallization of the phenocrysts. In recent work in growing certain synthetic minerals, he has found that under conditions of slow crystallization a mineral will form completely, but with rapid crystallization lineage structure develops and uncrystallized glass blebs remain in the crystal (in a pattern similar to that shown in the phenocryst in Fig. 2).

This hypothesis would seem to explain why such inclusions are far more common in the rapidly-chilled glassy rocks than in more crystalline volcanic rocks. It would also explain the occurrence of the glass sometimes found in the center of the phenocrysts as true inclusions not connected with the inward projections of the glassy groundmass of the rock.

However many of the so-called inclusions show strong evidence of having formed in a late stage and appear to be an extension of the glass base of the groundmass into the phenocryst. Coombs has noted a similar appearance in the Nisqually glacier flows on Mount Rainier: "The phenocrysts have ill-defined margins which seem to grade insensibly into the groundmass. On further examination almost all the feldspars showed this effect; an amount out of all proportion to the percentages expected in tapering wedges. Williams has figured a similar effect from the Mt. Harkness lavas."¹⁰ Williams has recently observed: "That the inclusions develop at a late stage is clear. Often I have noticed that the glassy base of a lava is traceable into the cores of feldspars."¹¹

It would appear that the phenocrysts had become more and more out of equilibrium with the molten material, and that violent ejection with accompanying fracturing of the phenocrysts assisted the hot and gas-rich residual liquid in working its way into and along the cracks of the crystals, with subsequent reaction. Retrograde boiling processes appear to have been at work.

It would be expected that these later reactions would be most effective along natural zones of weakness in the crystal, so that it is difficult to differentiate between true lineage structure effects and later corrosion. Also, many phenocrysts are crowded with glass inclusions that show no discernible arrangement within the feldspar.

Coombs¹² has suggested that more than one type or generation of glass may occur. The Haylmore pumice offers no positive optical evidence here, for no differences in refractive index of the glass have been

⁹ Buerger, M. J., Personal communication, Dec. 27, 1946.

¹⁰ Coombs, H. A., The geology of the Mount Rainier National Park: *Univ. Washington Publ. Geol.*, **3**, No. 2, 180 (1936).

¹¹ Williams, Howel, Personal communication, Jan. 20, 1947.

¹² Coombs, H. A., Personal communication, Nov. 5, 1946.

observed. Nevertheless, there are examples of zoned phenocrysts in which a glass-filled zone is surrounded by a clear shell of more albitic plagioclase, with some embayment of the outer zone by the glassy groundmass; in such cases an earlier and a later formation of glass seem to be represented. In the many phenocrysts which are completely riddled with glassy inclusions, more than one process may well have been at work; but the formation of glass has been so extensive that evidence of the nature of these processes has been destroyed.

The following analysis of the Haylmore pumice was made in the Rock Analysis Laboratory of the University of Minnesota, E. V. Kerr, Analyst; Lee C. Peck, Chief Chemist:

SiO ₂	66.05
Al ₂ O ₃	15.48
Fe ₂ O ₃	1.31
FeO	2.00
MgO	1.38
CaO	3.30
Na ₂ O	4.30
K ₂ O	2.21
H ₂ O+	2.25
H ₂ O-	0.36
TiO ₂	0.50
P ₂ O ₅	0.23
MnO	0.07
Total	99.44

The calculated norm is as follows:

q	23.34
or	12.69
ab	36.16
an	15.29
c	0.51
en	3.50
fs	1.98
mt	1.86
il	0.91
ap	0.34
C.I.P.W.: 1424.	

The chemical analysis reaffirms the dacitic composition of the rock. This analysis is very similar to that of a dacite pumice from Sun Creek Canyon, Crater Lake,¹³ and further suggests the possibility of regional kinship.

¹³ Williams, Howel, The geology of Crater Lake National Park, Oregon, *op. cit.*, p. 152.

ACKNOWLEDGMENT

The writer gratefully acknowledges the valuable information and suggestions given by Professor M. J. Buerger of the Massachusetts Institute of Technology, Professor Howard A. Coombs of the University of Washington, and Professor Howel Williams of the University of California. Thanks are also due J. M. Cummings of the British Columbia Department of Mines for information concerning the Meagher Mountain volcanic vent, Dr. Lee C. Peck of the Rock Analysis Laboratory of the University of Minnesota for cooperation in connection with the chemical analysis, and Dr. John S. Stevenson of the British Columbia Department of Mines for a critical reading of the manuscript.

NICKELIAN EPSOMITE FROM NORTH AUCKLAND, NEW ZEALAND

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ABSTRACT

The optical and chemical properties of nickelian epsomite are described, and the physical constants are found to coincide with the appropriate curves in the epsomite-morenosite series. Attention is drawn to the deficiency in the water content, which, it is believed, results from the relatively loose nature of the bonds holding one of the molecules of water within the structure. The action of sulfuric acid derived from oxidizing pyrrhotite on serpentinite and pentlandite is suggested for the occurrence.

OCCURRENCE

The mineral described herein as nickelian epsomite was found as efflorescent patches of pale blue-green crystals on freshly broken surfaces of serpentinite in Gordon's Quarry, Kaukapakapa, about thirty miles northwest from Auckland City. The host rock is a serpentinite of the dunite type enclosing scattered, irregularly shaped grains of pentlandite-bearing pyrrhotite, and it is on these sulfidic areas that nickelian epsomite has crystallized. In the same quarry nickel-free epsomite¹ was also observed at other points on the serpentinite surface, but in these instances only traces of nickel were detected in the associated pyrrhotite.

Nickelian epsomite formed clusters of minute needle-like crystals that rarely exceeded 4 mm. in length, and averaged very much less; the areas covered by these efflorescences were about nine to twelve inches square.

CHEMICAL PROPERTIES

The mineral is very soluble in cold water to give a slightly acid solution with a very bitter and astringent taste, and qualitative chemical tests showed that magnesium, nickel, the sulfate radical, and water were the only constituents present; the absence of aluminum and particularly iron, is noteworthy. Water is evolved from the crystals very readily and the greater percentage of it is lost below 105° C. After heating to about 300° C. a pale yellow to greenish-yellow anhydrous powder remains.

An analysis of a very small quantity of the mineral, made by Mr. F. T. Seelye, is given in Table 1. In a second determination of total water by fusion with sodium carbonate (Gooch method) Mr. Seelye obtained a

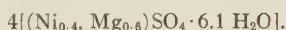
¹Optics determined for this mineral are as follows: $\alpha=1.434$, $\beta=1.456$, $\gamma=1.461$; $\gamma-\alpha=0.027$. $2V=52^\circ$. Microchemical tests proved the absence of both iron and nickel.

TABLE 1. ANALYSIS OF NICKELIAN EPSOMITE

NiO		12.22%	
MgO		9.81	
SO ₃		33.05	
H ₂ O	{ at 105° C.	38.78	44.92
	{ 105° — 225° C.	1.62	
	{ > 225° C.	4.52*	
			100.00%

* Determined by difference.

figure of 44.8 per cent. Since the heptahydrates of magnesium and nickel sulfates contain four molecules to the unit cell (Westenbrink, 1926; Cardoso, 1926) the analysis above may be recalculated and expressed approximately as follows:



PHYSICAL PROPERTIES

Aggregates of acicular crystals of nickelian epsomite are pale blue-green in color (16V in Radde's color scale), but individual crystals appear to be quite colorless when immersed in refractive index media. Crystals are elongated parallel to the Y vibration direction of the refractive index ellipsoid and in addition they show a marked flattening parallel to the plane containing the X and Y directions. Hence the majority of the crystals are positively elongated whereas only relatively few are found to be fast in that direction. The range of refractive indices determined by the immersion method in sodium light is as follows:

$$\begin{aligned}\alpha &= 1.446 - 1.448 \pm 0.002 \\ \beta &= 1.470 \\ \gamma &= 1.472 - 1.474 \\ \gamma - \alpha &= 0.026\end{aligned}$$

Since the mineral readily loses water when it is heated it was not considered desirable to mount it in Canada balsam for immediate optic axial angle determination; instead a few needles were cemented in the cold state on a glass slip with clarite. The average of a number of measurements of the angle $2V$ was $47^\circ \pm 2^\circ$; there is a negative optic sign and no dispersion.

One fair cleavage was noted normal to the direction of prominent flattening, (010) cleavage, and a poor cleavage, probably (011), was seen to bevel the ends of the acicular crystals in a number of instances. Details of the optic orientation are shown diagrammatically in Fig. 1.

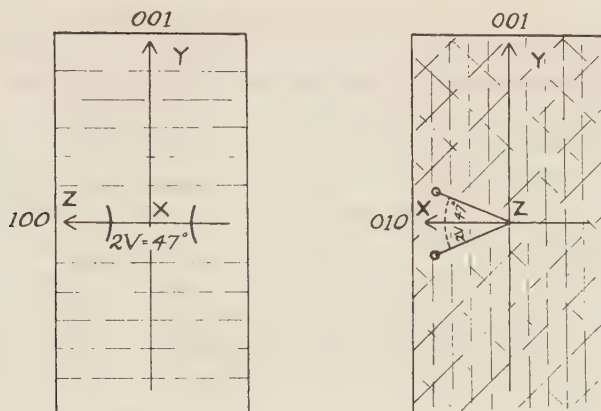


FIG. 1. Optical orientation of nickelian epsomite.

DISCUSSION OF PROPERTIES

There are two significant points in connection with this mineral that require comment, and it is necessary that they should be considered at the same time.

(a) The number of water molecules is less than the theoretical figure for a heptahydrate formula, but distinctly above that required for the hexahydrate, and

(b) In spite of the low figure for water the physical constants correspond almost exactly with those that would be expected for an intermediate member of the epsomite ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$)-morenosite ($\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$) series.

Few analyses of either morenosite or any intermediate members of the epsomite-morenosite series appear in the literature available to the writer, and even fewer are accompanied by refractive index data. However deficiency in water appears to be a feature of a number of these analyses; for example the formula $\text{NiSO}_4 \cdot 6.5 \text{H}_2\text{O}$ was derived from an analysis of morenosite from Valtournanche by Cavinato (1938), and in an epsomite² from Rakos,³ Slovakia, Zsivny (1915) also found water to be slightly less than the normal seven molecules. Again Simpson (1920) has described cobaltian epsomite from Parkerville, West Australia, with only 5.7 molecules of water, whereas Walker and Parsons (1927) have found that the compositions of crystals of magnesium sulfate from lake

² Since 1.78 per cent of MnO was found therein the mineral is probably the variety fauserite.

³ Formerly Rákobánya in Hungary.

deposits near Oroville, Washington, and from Ashcroft, British Columbia, show a range of from six to seven molecules of water depending upon atmospheric conditions. On the other hand, a few analyses of

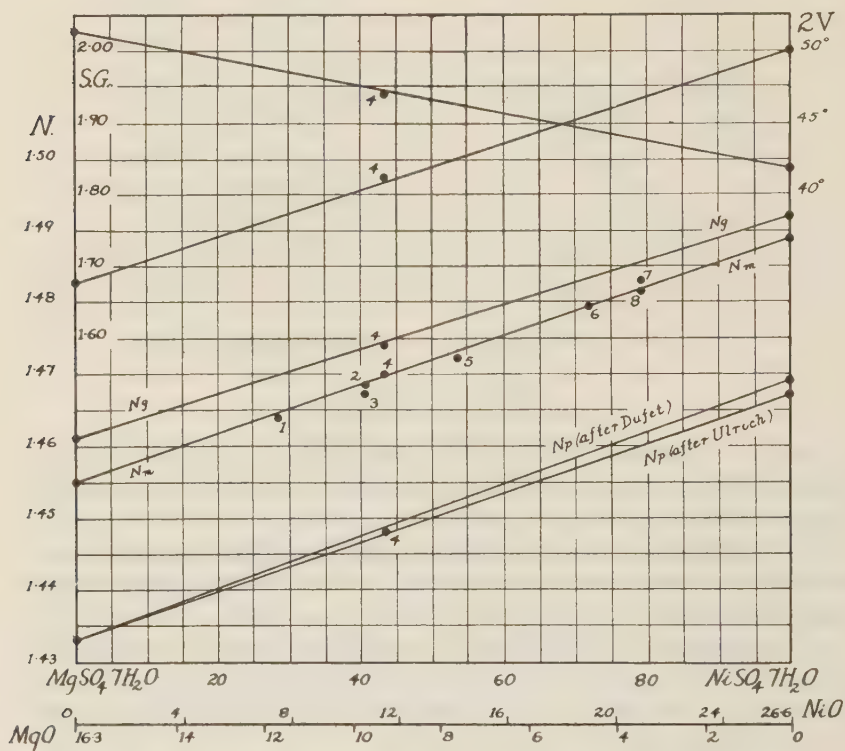


FIG. 2. Variations in composition and physical properties in the epsomite-morenosite series.

Key to analyses in Fig. 2:

1. 71.65 per cent $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. Dufet (1878), compound no. 1.
2. 59.3 per cent $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. Dufet (*loc. cit.*), compound no. 2; β calculated.
3. As in 2, but β observed.
4. Data for Kaukapakapa mineral.
5. 46.1 per cent $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. Dufet (*loc. cit.*), compound no. 3.
6. 28.05 per cent $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. Dufet (*loc. cit.*), compound no. 4.
7. 20.9 per cent $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. Dufet (*loc. cit.*), compound no. 5; β observed.
8. As in 7, but β calculated.

members of the epsomite-morenosite series are available that give no indication of any deficiency in the number of molecules of water (Ulrich, 1921; Kokta, 1930), and in some instances the analyses are unsatisfac-

tory in that water has been determined only by difference (Jirkovsky, 1928, pp. 59-60).

In view of the care taken to determine the full amount of water and also the ease with which nickel and magnesium sulfates yield water, it might be suggested that the deficiency in the case of the New Zealand mineral at least, is to be attributed to the presence of an intermediate member of a hexahydrate solid solution series. Certainly a lower hydrate of magnesium sulfate, though rare, does occur in nature, *viz.* hexahydrate, and it might be expected to form an ionic substitution series with the corresponding hexhydrate of nickel sulfate,⁴ but the optical properties, particularly optic orientation, of this compound would be distinct enough from those recorded for the New Zealand mineral to permit differentiation.

In Fig. 2 of this paper curves for the variation of refractive indices, optic axial angle, and specific gravity with composition in the epsomite-morenosite series have been drawn up. The data for epsomite⁵ are those quoted by Winchell (1933) and Larsen and Berman (1934), whereas in the case of morenosite the values for α , β , γ , and $2V$ are those determined by Dufet (1878, p. 882) for the pure salt. It should be noted that other determinations for the nickel salt made by Topsoe and Christiansen (*vide* Ulrich, 1921, p. 124) gave a distinctly lower figure for the refractive index of the X vibration direction, *viz.* 1.4669, than that recorded by Dufet; this value is represented by the lowermost point for morenosite on the N_p line. Dufet's determinations, both observed and calculated, of the refractive indices for the Y vibration direction in a number of the synthetic intermediate members of the series have been plotted in Fig. 2, and a straight line variation results.

When the analysis of the New Zealand mineral is recalculated to seven molecules of water and the physical constants plotted on Fig. 2, coincidence is such that the existence of a member of a hexahydrate solid solution series therein seems to be out of the question. Further, careful inspection of the material when mounted in an oil of refractive index 1.470 did not reveal the existence of a second, but distinct, crystal-line phase. It would seem then that the loss of a small percentage of water from the heptahydrate does not necessarily cause any appreciable variation in the optical properties characteristic of members of that series;

⁴ $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ is dimorphous, crystallizing from solution at about 35° C. in tetragonal forms, or at about 60° C. in the monoclinic system.

⁵ The value of 1.751 given by Ford (1932) for the specific gravity of epsomite appears to be an error; it should also be noted that the heptahydrate of magnesium sulfate is dimorphous and the specific gravity of the normal orthorhombic form is 1.677, whereas the figure for the monoclinic modification is 1.691 (Doelter and Leitmeier, 1929, p. 20).

or alternatively, since loss of water is almost invariably found to cause a rise in the refractive indices of minerals, loss of water from nickelian epsomite, while still retaining orthorhombic disphenoidal symmetry of the stable heptahydrate, does not bring about any easily measurable rise in the value of the refractive indices. Any pronounced change in optical properties then is only to be expected when one molecule of water has been removed, and the salt recrystallizes with monoclinic (or possibly tetragonal) symmetry of the true hexahydrate.

In an ionic compound such as $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$, six of the water molecules are in octahedral groups surrounding each nickel ion, but the seventh is isolated and is linked not to cations, but instead to oxygen ions of the SO_4 groups, and to other water molecules (*vide* Beevers and Schwartz, 1935). The function of the first six water molecules is clearly to co-ordinate the cations and to promote a stable structure by enlargement of the effective radius of the cations, whereas the position of the seventh water molecule appears to be of less importance but it is certainly more vital than the role of water in zeolites, since it is accommodated in small cavities in the structure and has developed hydrogen bonds. It is suggested therefore, that the structure of nickelian epsomite could retain its stability, even after the loss of some of the "seventh" water molecules, and although this would result in a deficiency of water, the heptahydrate symmetry would be retained.

ORIGIN OF EPSOMITE

The close association of the two sulfates, nickelian epsomite and epsomite, with pyrrhotite clearly suggests that the sulfide has been a vital factor in their formation, and there seems to be little doubt that the processes that have been formative here are similar to those that produced heavy efflorescences of morenosite on pyrrhotite-pentlandite ores at Sudbury (Coleman, 1913, p. 20). Processes that involve oxidation of pyrrhotite to give ferric sulfate and sulfuric acid are well known and need no elaboration here; the sulfuric acid so derived has apparently reacted with the readily soluble serpentinite and with the pentlandite in the pyrrhotite giving sulfates of nickel, magnesium, and iron. It is assumed that the primary nickel is confined to the sulfide since qualitative tests indicate that only strong traces of nickel occur in the serpentinite itself.

The virtual absence of iron from these salts in view of its relative abundance in the source materials is interesting. However, the separation of iron from nickel during oxidation of sulfide ores is well known (Emmons, 1917, p. 460), and arises solely from the rapidity with which

ferrous sulfate is oxidized to basic ferric sulfate, and ultimately hydrolyzed to limonitic products; on the other hand, nickelous salts are stable and do not oxidize or hydrolyze in air as iron compounds do. Thus iron will remain behind near its source whereas nickel is removed in solution and permitted to crystallize in forms uncontaminated by iron; magnesium will behave in a similar manner to that of nickel.

NOMENCLATURE

According to Mellor (1936, p. 454) the heptahydrate of nickel sulfate was termed pyromeline by F. von Kobell, but Casares' name—morenosita—after St. Moreno, has definite priority. Later Pisani (*vide* Doelter and Leitmeier, 1929, p. 612) gave the name pyromeline to a stalactitic mineral from Zermatt which was an intermediate member of the $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ - $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ series with a composition similar to, but slightly richer in nickel than, the New Zealand mineral described herein. Therefore, in view of possible ambiguity the term pyromeline should not be employed but instead the terms nickelian epsomite and magnesian morenosite should be used to denote intermediate members of the series.

ACKNOWLEDGMENT

This work was undertaken in the Geology Department of the University of Otago, Dunedin, New Zealand.

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CHARTS FOR CORRELATION OF OPTICAL PROPERTIES WITH CHEMICAL COMPOSITION OF SOME COMMON ROCK-FORMING MINERALS*

GEORGE C. KENNEDY, *Society of Fellows, Harvard University.*

The following group of charts has been assembled from sources as noted below. The data, though the best now readily available, are in part subject to correction. Persons using these charts are requested to note any errors that come to their attention.

The feldspar curves were compiled by F. C. Calkins and modified by Hess in the region of the highly calcic plagioclases.

Curves for the high-magnesium end of the orthopyroxene diagram are after curves by H. H. Hess, published in *American Mineralogist*, **25**, 271-285 (1940). Curves for the high-iron end have been constructed by the writer from inferior data and are not of the same reliability as Hess's data. The curve for 2V is from Hess.

Curves for the diopside-hedenbergite series and for the diopside-johannsenite-hedenbergite triangular diagram are after unpublished curves drawn by Waldemar T. Schaller.

Curves for clinoenstatite-clinohypersthene-diopside-hedenbergite are taken from a diagram published by Toru Tomita in *Jour. Shanghai Sci. Inst.*, Sec. 2, **1**, 46 (1934), and modified by the writer in the diopside-hedenbergite range of the system using Schaller's data.

The olivine curves are from data obtained on synthetic minerals, by N. L. Bowen and J. F. Schairer, published in *American Journal of Science*, **29**, 197 (1935). The curve for 2V has been calculated from these data by Clyde Wahrhaftig.

Curves for the calcite group minerals are after unpublished curves constructed by Clarence S. Ross.

Data for the garnet triangular diagrams were compiled by Michael Fleischer and published in *American Mineralogist*, **22**, 751-759 (1937).

Grateful acknowledgment is due the contributors to this compilation. Professors E. S. Larsen, Jr., Clifford Frondel and Cornelius S. Hurlbut have carefully checked the diagrams and made many suggestions in their presentation.

* Contribution from the Department of Mineralogy and Petrography, Harvard University, No. 285.

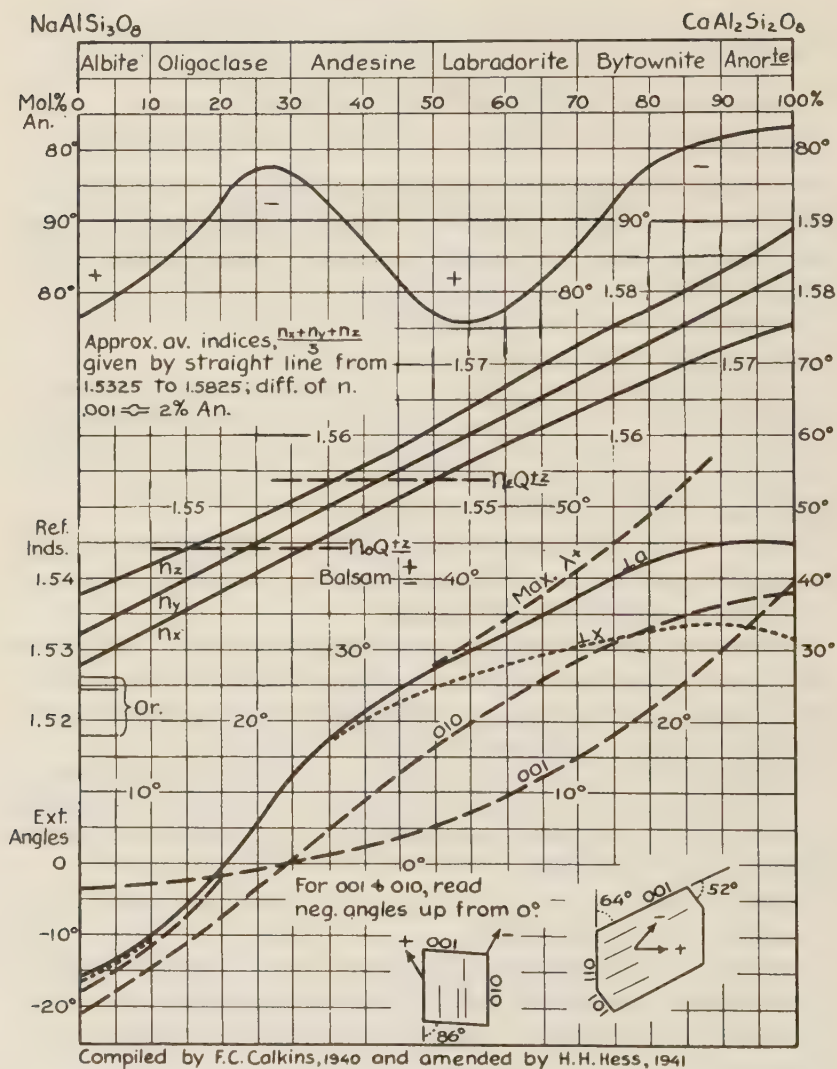


FIG. 1. Variation of optical properties with chemical composition in the plagioclase-feldspar group.

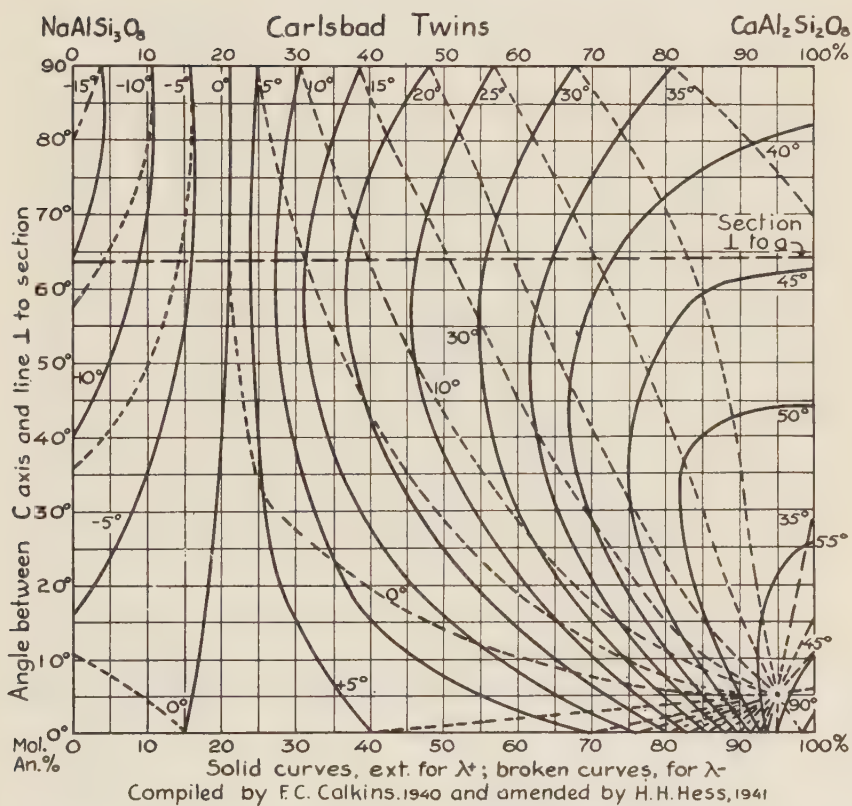


FIG. 2. Variation of optical properties with chemical composition in the plagioclase-feldspar group.

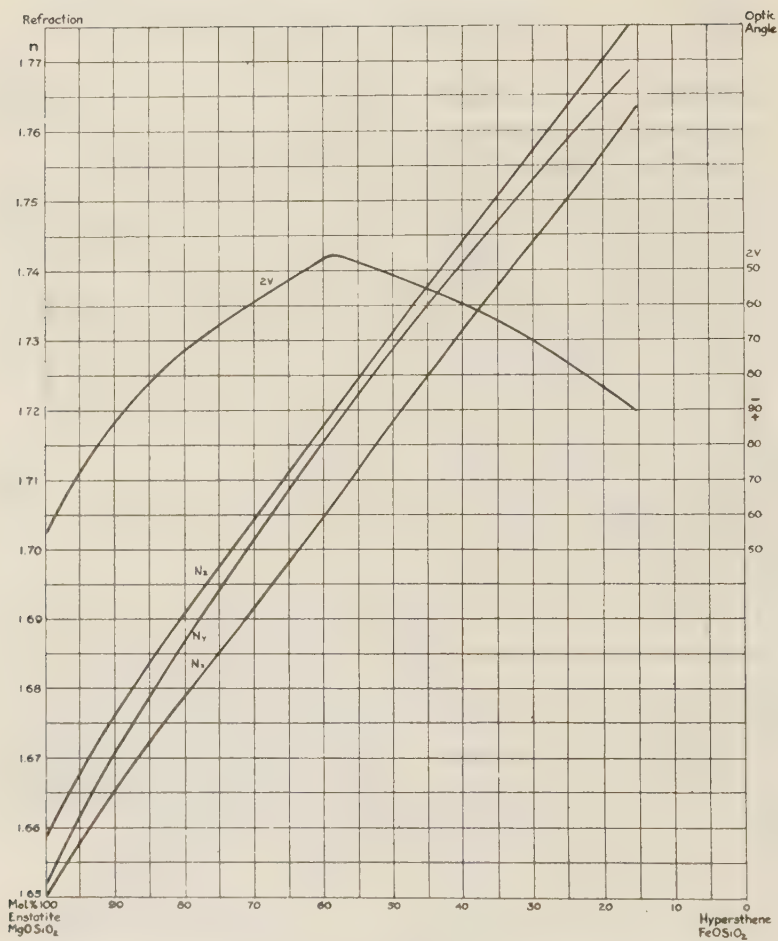


FIG. 3. Variation of optical properties with chemical composition in the enstatite-hypersthene series.

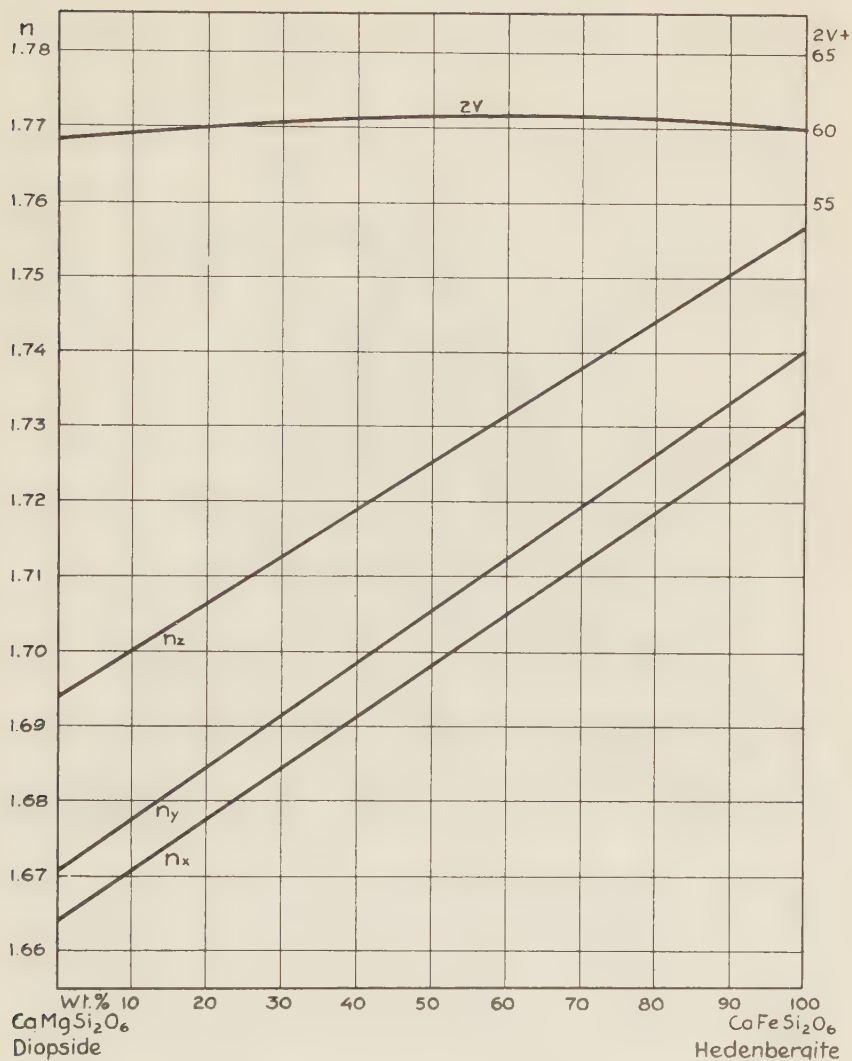


FIG. 4. Variation of optical properties with chemical composition in the diopside-hedenbergite series.

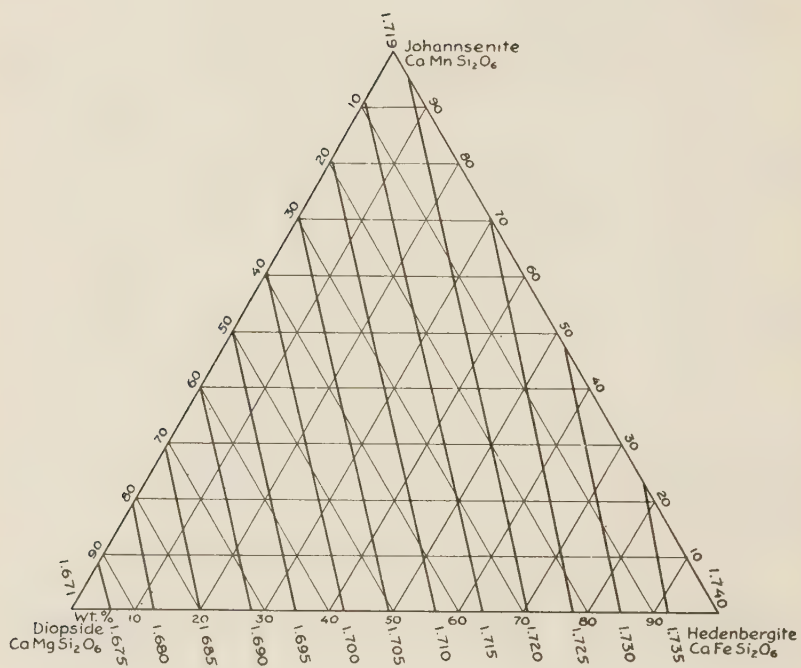


FIG. 5. Variation of N_y with composition of diopside hedenbergite-johannsenite system.

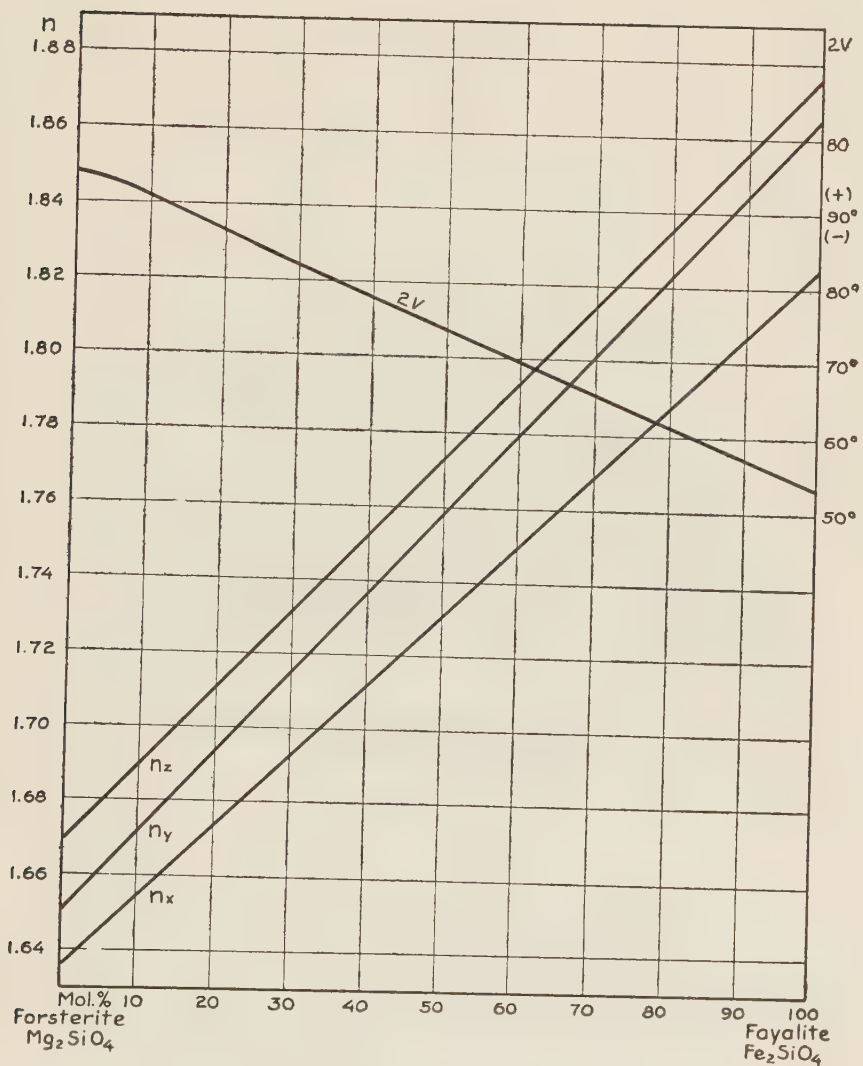


FIG. 6. Variation of optical properties with composition in the forsterite-fayalite series.

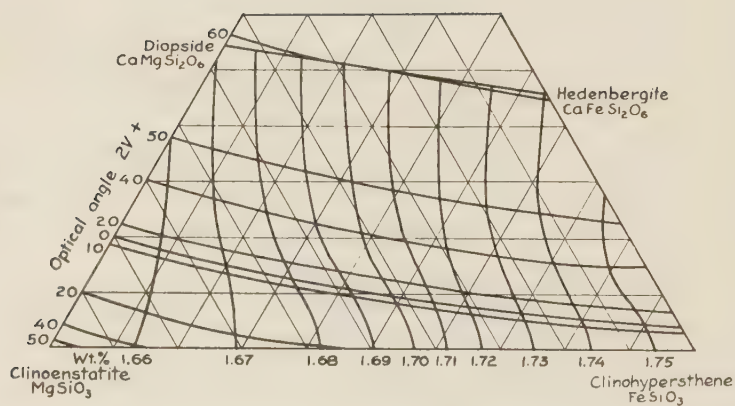


FIG. 7. Variation of N_y and $2V$ with composition in the clinoenstatite-clinohypersthene-diopside-hedenbergite system.

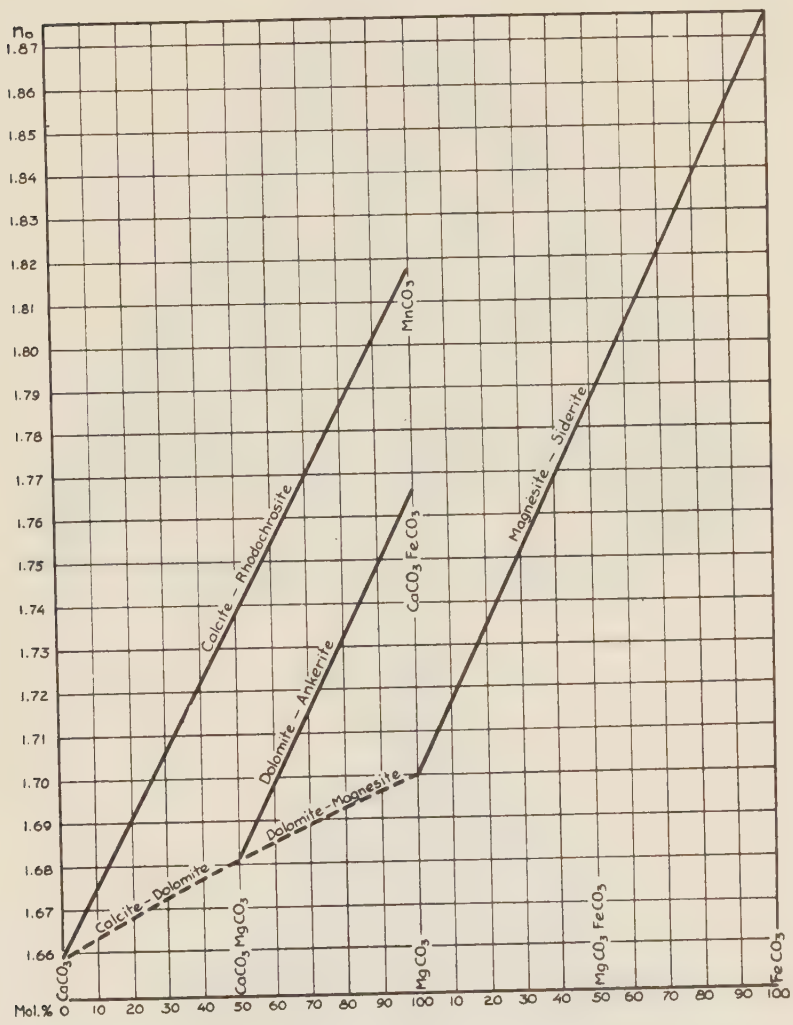


FIG. 8. Variation of N_o with composition in calcite group minerals.

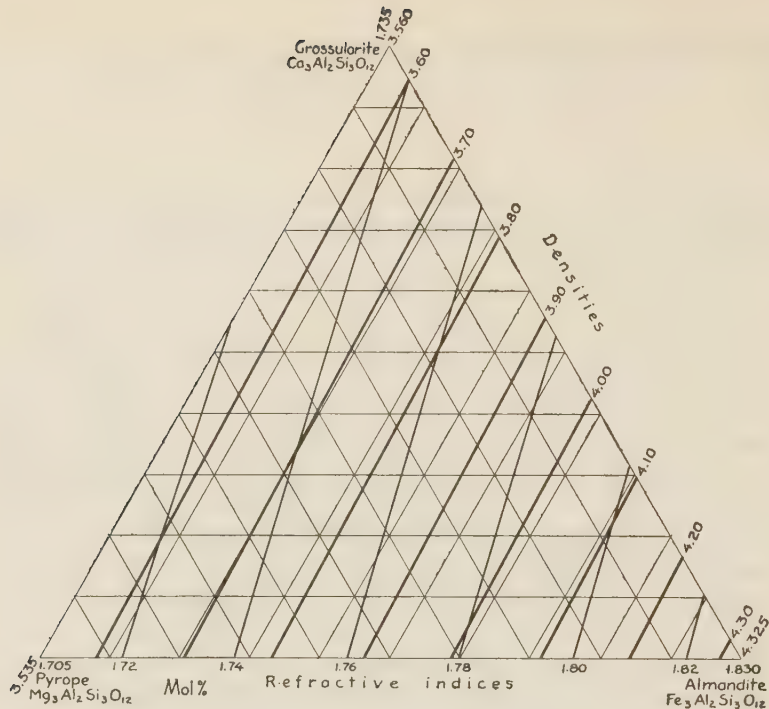
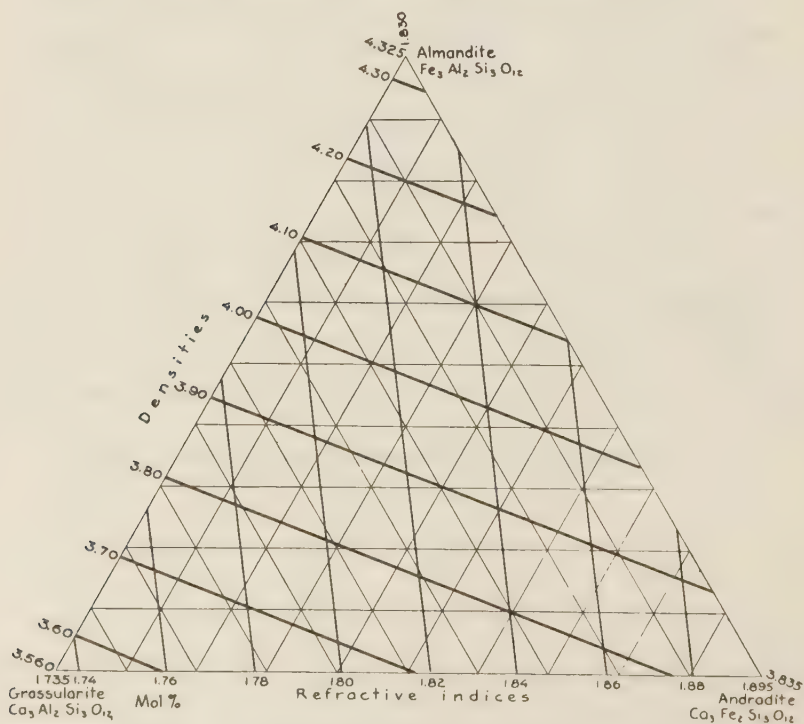
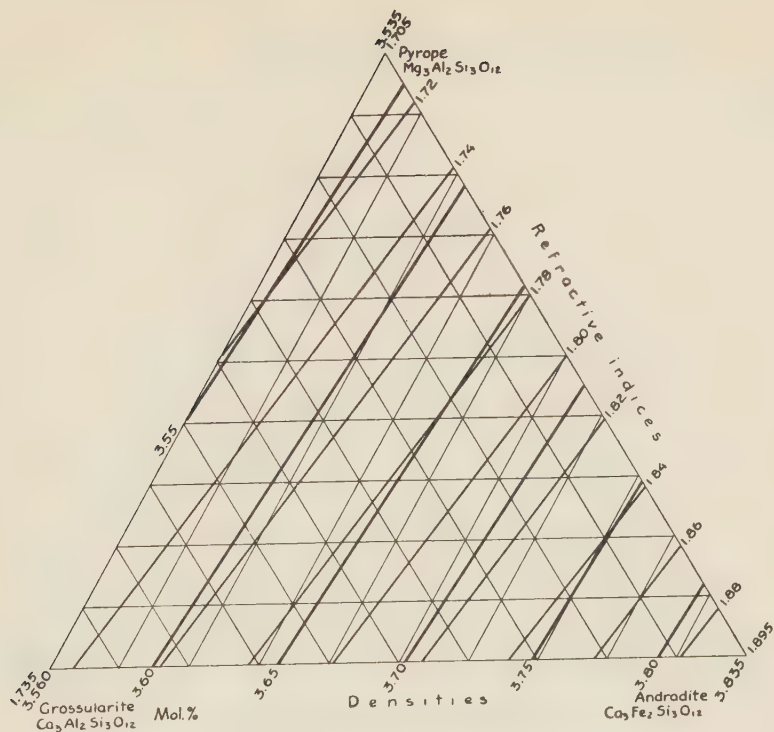


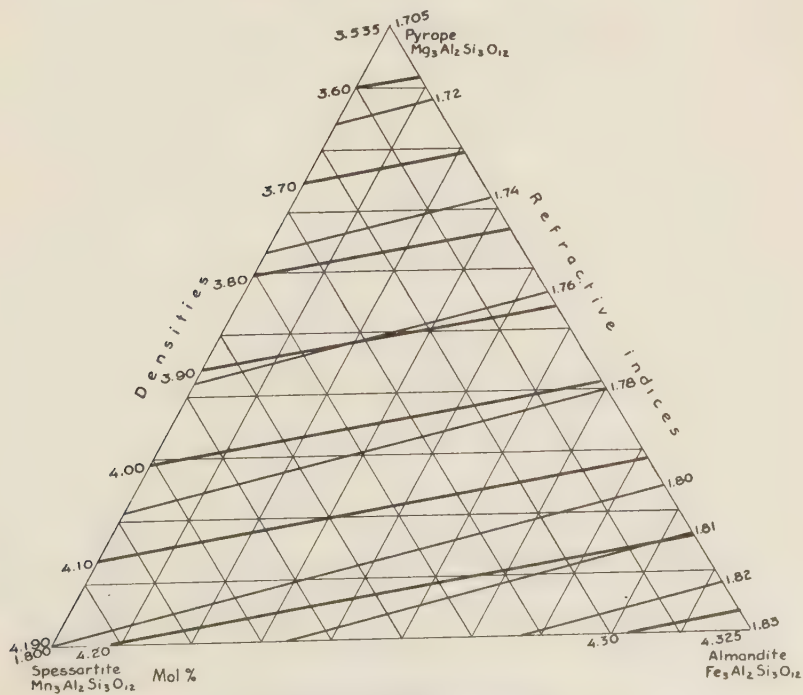
FIG. 9. Variation of physical properties with chemical composition in the garnet group minerals. a. Grossularite, pyrope, almandite.



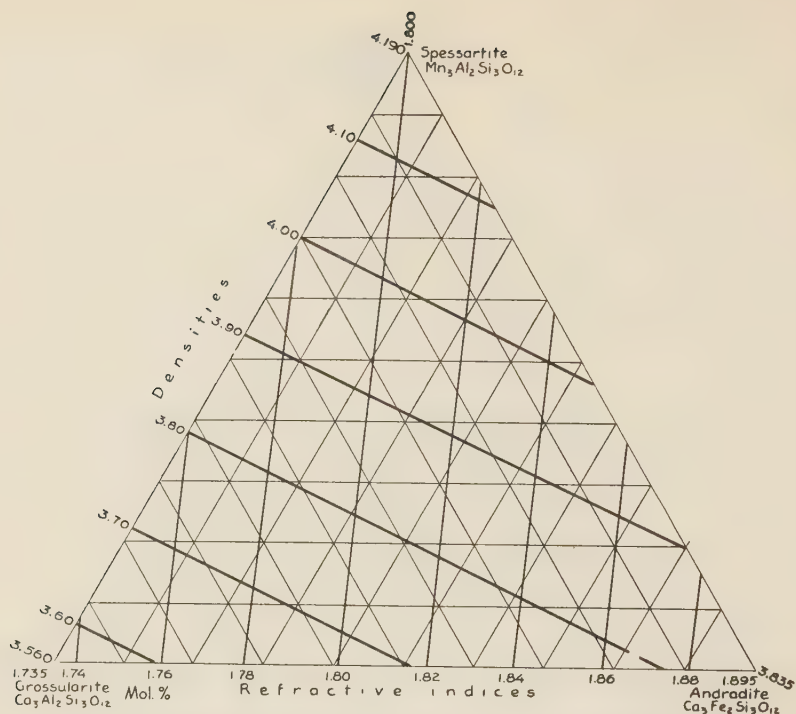
b. Almandite, grossularite, andradite.



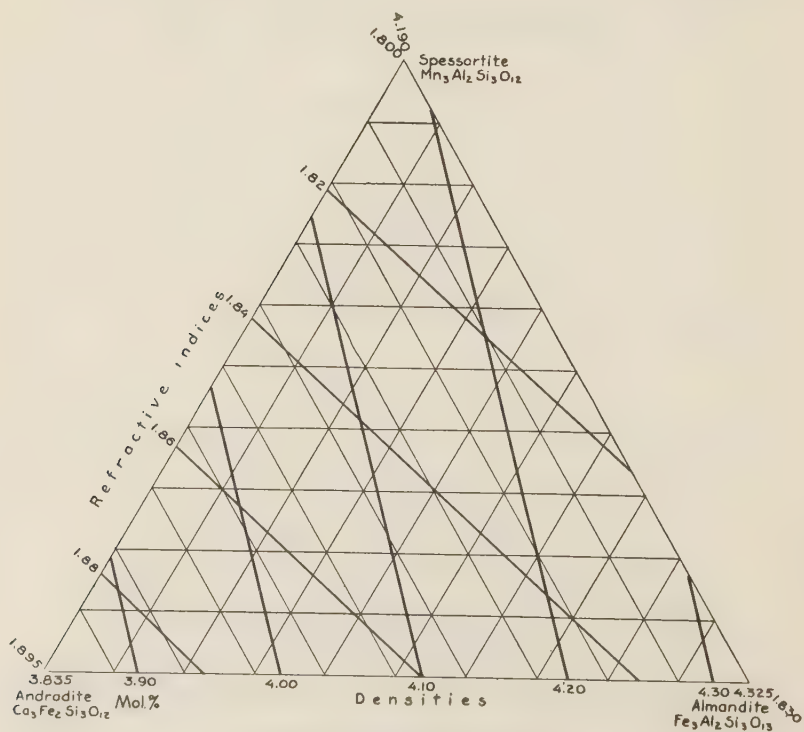
c. Pyrope, grossularite, andradite.



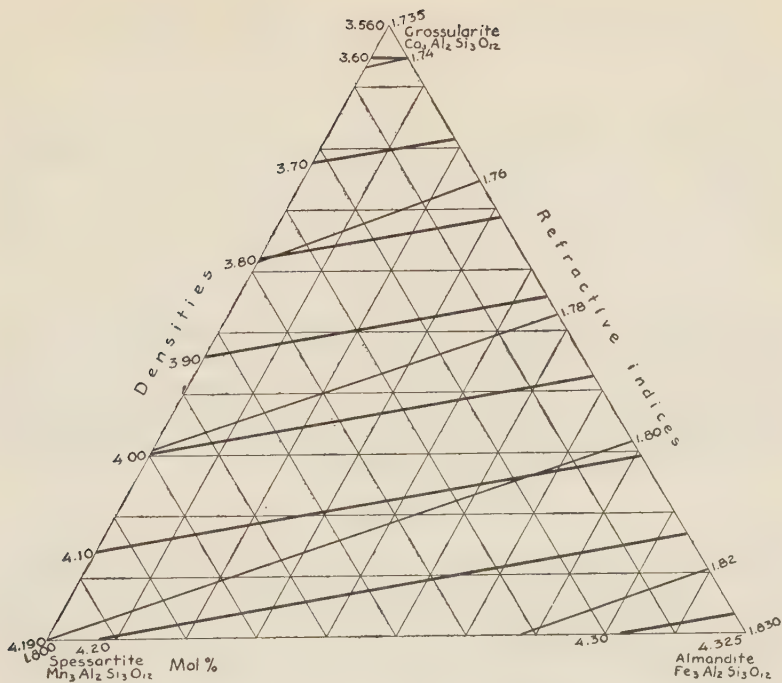
d. Pyrope, spessartite, almandite.



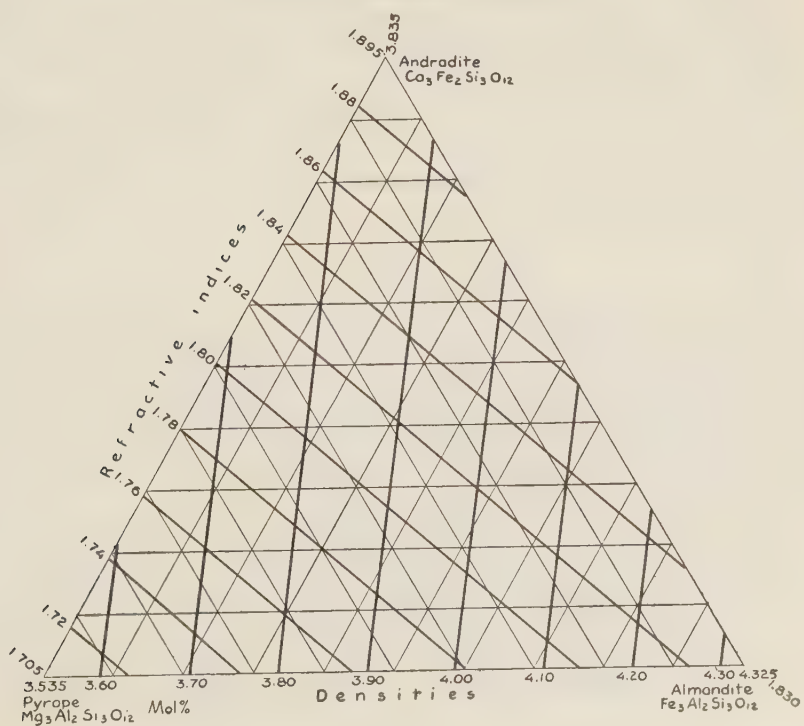
e. Spessartite, grossularite, andradite.



f. Spessartite, andradite, almandite.



g. Grossularite, spessartite, almandite.



h. Andradite, pyrope, almandite.

PHOSPHATE MINERALS IN THE CASTLE DOME COPPER DEPOSIT, ARIZONA

N. P. PETERSON*

ABSTRACT

Several phosphate minerals occur in the Castle Dome copper deposit near Miami, Arizona, some of which are of special interest because of their uncommon occurrence in deposits of this type. They comprise apatite, turquoise, wavellite, metatorbernite, and libethenite. Apatite is a primary constituent of the quartz monzonite host rock. Turquoise and libethenite are probably supergene minerals, whereas wavellite and metatorbernite may possibly have been introduced by late hydrothermal solutions.

INTRODUCTION

The Castle Dome copper deposit is in the Globe-Miami mining district, Gila County, Arizona, five miles west of Miami. The Castle Dome open-pit mine, on the south slope of Porphyry Mountain, is owned and operated by the Castle Dome Copper Company, Inc. It was developed by the owners as a war project, and since June 1943 has produced about 4,000,000 pounds of copper per month.

A detailed study of the Castle Dome deposit and the surrounding area was undertaken by the U. S. Geological Survey in 1943. A complete description will be published by the Survey at a later date.

The writer is indebted to Jewell J. Glass for her work on the properties of the libethenite and for checking the identification of the other minerals described. He wishes also to thank the Castle Dome Copper Company for permission to publish this paper.

THE COPPER DEPOSIT

The copper deposit is of the disseminated or "porphyry" type. The host rock is a body of quartz monzonite intruded into pre-Cambrian schists and quartzites and possibly into the lower part of the overlying Paleozoic strata. The quartz monzonite is much older than the copper mineralization which is probably genetically related to the Schultze granite south of the deposit. The Schultze granite is of probable late Cretaceous or early Tertiary age.

The most important hypogene sulfides in order of abundance are pyrite, chalcopyrite, and molybdenite. They occur in, or associated with, a set of narrow, closely spaced, generally parallel, quartz veins striking east-northeast and dipping steeply southward. The only other hypogene

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sulfides, sphalerite and galena, are present in relatively small amounts. They occur accompanied by some quartz, pyrite, and chalcopyrite in veins deposited along faults and major fractures many of which cut across the pyrite, chalcopyrite, and molybdenite veins. The sphalerite-galena veins are banded as if they had been deposited in open fractures, and commonly the vein minerals do not completely fill the fractures. They apparently represent a slightly younger phase of mineralization than the pyrite, chalcopyrite, and molybdenite veins. Small amounts of barite and fluorite, deposited on the walls of open fractures, were also late hypogene minerals.

The mineralizing solutions altered the quartz monzonite bordering the veins to quartz and sericite and caused general argillic alteration of the plagioclase throughout the mineralized area.¹

Although the ore body is partly the result of higher than average grade copper metallization associated with several gently dipping diabase sills intruded into the quartz monzonite, supergene enrichment played an important role in the formation of the ore. In the upper part of the ore body, chalcopyrite is partly replaced by chalcocite; but the enrichment has not progressed so far that pyrite is replaced except to a very minor extent in a few places. A little covellite is present throughout the the chalcocite zone, but it is generally abundant near the top where it formed by oxidation of chalcocite. Oxidized copper minerals, including malachite, azurite, cuprite, and native copper, occur sparsely throughout the leached and chalcocite zones. Turquoise is also fairly common in these zones.

PHOSPHATE MINERALS

In addition to the common sulfides and supergene minerals, a number of phosphates occur in the deposit, some of which are of special interest because of their uncommon occurrence in this type of ore body. They comprise apatite, turquoise, wavellite, metatorbernite, and libethenite. Apatite and turquoise are widely distributed; whereas wavellite, metatorbernite, and libethenite are present only in certain parts of the mine.

Apatite.—Apatite is an accessory constituent of the quartz monzonite host rock in which it occurs associated with biotite in a manner common in rocks of this kind. No apatite introduced by hydrothermal solutions has been recognized. It is mentioned here as a possible source of the phosphate ion in the other minerals.

The phosphate content of the fresh and hydrothermally altered quartz monzonite, as determined by chemical analysis, is shown as follows:

¹ Peterson, N. P., Gilbert, C. M., and Quick, G. L., Hydrothermal alteration in the Castle Dome copper deposit, Arizona: *Econ. Geol.*, **41**, 820-840 (1946).

Sample No	1	2	3	4	5
P ₂ O ₅	.23	.19	.13	.13	.08%
Samples 1 and 2. Unaltered quartz monzonite					
Sample 3. Altered quartz monzonite (clay phase)					
Sample 4. Altered quartz monzonite (clay-sericite phase)					
Sample 5. Altered quartz monzonite (quartz-sericite phase)					

Apatite is the only phosphate mineral that was recognized in thin sections of the analyzed rocks. On the basis of these analyses, the fresh rock contains about 0.5 per cent apatite. From 40 to 60 per cent of it is destroyed by hydrothermal alteration. Apatite is generally considered to be a fairly stable mineral but is soluble in acids and is undoubtedly dissolved by supergene solutions to some extent. It could therefore serve as a source of phosphate ion in the formation of supergene minerals.

Wavellite, $3Al_2O_3 \cdot 2P_2O_5 \cdot 13(H_2O, HF)$.—Wavellite is restricted largely to a relatively small elongate area, which trends parallel to the mineralized veins, in the central part of the ore body. It appears to be localized along fractures dipping moderately north to northwestward, which cut the steeply southward dipping veins at approximately right angles. Where open spaces occur along the fractures, wavellite forms encrustations on the walls and on earlier minerals deposited on the walls of the fractures. The crusts consist of coalescing hemispherical masses up to 4 mm. in diameter with radiating internal structure. The surfaces of the hemispheres glisten with light reflected from countless crystal faces. Some of the crusts are clear and colorless, others are gray, yellow or pale-green. In the weathered zone the crusts are usually translucent white or may be stained by iron.

Metatorbernite, $CuO \cdot UO_3 \cdot P_2O_5 \cdot 8H_2O$.—Metatorbernite was first recognized as an artificial mineral by Hallimond.² He obtained it by dehydration of torbernite. The first inversion of torbernite in contact with water occurs at 75° C. It is accompanied by a marked change in optical properties and a loss of four molecules of water. A second inversion takes place at 130° C. with further loss of water. The two products were designated by Hallimond as metatorbernite I and II, respectively. He was unable to bring about a reversal of the transformations. Metatorbernite I was later recognized as a natural mineral from Spain and Cornwall by N. L. Bowen.

² Hallimond, A. F., The crystallography and dehydration of torbernite: *Mineral. Mag.*, 17, 326-339 (1916).

The metatorbernite in the Castle Dome deposit was, almost without exception, deposited on wavellite crusts. It occurs as very thin, rectangular, bright-green plates which are usually arranged in tiny rosettes of extraordinary beauty. In some places the crystals form a moss-like mat covering the wavellite crusts. Examined in immersion oils, the crystals are seen to be rectangular or to show rectangular cleavage. Plates lying face up show no birefringence and have an index of refraction of 1.623. The other index is slightly higher. Tilted plates show anomalous purple to violet interference colors and slight pleochroism from yellowish-green to greenish-blue. These properties identify the mineral as metatorbernite I described by Hallimond.³ Bowen⁴ studied the abnormal birefringence of metatorbernite I and found the mineral to be positive for the red end of the spectrum, negative for the blue end, and isotropic in green light at about 515 $\mu\mu$.

The metatorbernite in the Castle Dome deposit is present only in very small amounts.

Libethenite, $4\text{CuO} \cdot \text{P}_2\text{O}_5 \cdot \text{H}_2\text{O}$.—Libethenite has been found only along fractures related to the Dome fault system which is the most prominent structural feature in the mine and consists of several normal faults trending northeastward through the central part of the ore body. Like wavellite and metatorbernite, it occurs only in open fractures where it forms crusts which are generally composed of tufts of small, emerald-green prisms or a drusy mat of acicular crystals. In some places discrete crystals, some so small they cannot be recognized without the aid of a lens, others up to 1.5 mm. long, lie flat on the rock surfaces.

Jewell Glass of the Geological Survey studied several specimens of the mineral and described its physical and optical properties as follows:

Crystals are usually short prisms two to five times as long as they are thick, passing into acicular forms grouped in radiating clusters, united in druses. A few crystals show pyramidal habit, resembling octahedrons. Cleavage is good prismatic, luster vitreous, color emerald to yellowish-green, transparent.

Optically the mineral is biaxial negative, $2V$ large, about 85° . An optic axis emerges normal to a prismatic cleavage or a side pinacoid and looks like the familiar optic axis on epidote grains. The plane of the optic axis is across the elongation. Twinning is observed occasionally. Dispersion is strong, $r > v$. The fragments are pale-green, pleochroism faint, seldom distinguishable. Extinction inclined 0° to 20° , greater than 20° in a few grains. Indices of refraction, average of several readings on each specimen, are: $\alpha = 1.703$, $\beta = 1.746$, $\gamma = 1.789$; $B = 0.086$, all ± 0.002 .

³ Hallimond, A. F., Metatorbernite I, its physical properties and relation to torbernite: *Mineral. Mag.*, **19**, 43-47 (1920).

⁴ Bowen, N. L., Abnormal birefringence of torbernite: *Am. Jour. Sci.*, 4th ser., **48**, 195-198 (1919).

The explanation for the anomalous inclined extinction of the libethenite may be revealed by its x -ray diffraction pattern which has not as yet been studied.

Wulfenite, $PbO \cdot MoO_3$.—Wulfenite, although not a phosphate, is discussed here because of its close association with libethenite. It was identified on fragments of rock broken by blasting and has not been seen in place. Instead of the usual tabular crystals, the Castle Dome wulfenite occurs as small, pointed, tetragonal prisms, rarely over 3 mm. long and generally much smaller, which are attached to fragments of rock that apparently formed the walls of open fractures. The surfaces of the rock, as well as some of the crystals, are coated with limonite, jarosite, canbyite, and malachite, all of undoubted supergene origin.

Turquoise, $CuO \cdot 3Al_2O_3 \cdot 2P_2O_5 \cdot 9H_2O$.—Turquoise is fairly widespread throughout the chalcocite zone and in the leached capping. A hard variety of gem quality occurs in small veinlets up to one-quarter inch thick and also in concretion-like masses up to one-half inch thick and several inches across. Its color ranges from sky-blue to bluish-green, the blue variety predominating. It is generally associated with clay minerals and sericite which form selvages on the veins and masses and are commonly included in the turquoise. Seen under the microscope, it is a microcrystalline aggregate having a mean index of refraction of 1.62.

Much more abundant than the hard blue turquoise is a soft chalky variety which occurs in small masses on the walls of open fractures or filling small fractures. The masses and seams were not formed by alteration or replacement of wall-rock but appear to be accumulations of material transported into the fractures either in solution or in suspension in ground water. Its color ranges from almost white to light-blue when dry and deeper shades of blue when moist. The powdered mineral in immersion oils is almost identical in appearance to the powdered hard variety and has the same index of refraction. When ignited, the powder turns brown, becomes soluble in acid and gives positive reactions for copper and phosphate ions. All gradations between the hard blue turquoise and the white chalky variety can be found.

A few specimens of a light-green, iron-bearing copper aluminum phosphate were found occurring in the same manner as the hard blue turquoise. It is a microcrystalline aggregate whose mean index of refraction is 1.67. Charles Milton of the Geological Survey identified the mineral as a ferrian turquoise. He reports as follows:

The light-green mineral is identified as turquoise rather than chalcosiderite. These two minerals are considered to be isomorphous with Al_2O_3 and Fe_2O_3 , respectively. The x -ray pattern is similar to that of turquoise from Lynch, Va., which presumably

would hardly be distinguishable from chalcosiderite. However, the density (2.75 by pycnometer) and indices of refraction indicate turquoise.

Employees of the mine have been permitted to dig turquoise on days when they were not on duty, and an amazingly large quantity of the mineral has been purchased by dealers in Globe and Miami. Unfortunately no record is available as to the amount of turquoise produced, but undoubtedly the Castle Dome mine is one of the most important sources of gem turquoise at the present time.

ORIGIN OF THE PHOSPHATES

Although some of the Castle Dome phosphates were probably formed by supergene processes, the lack of valid evidence of supergene origin in the case of wavellite and metatorbernite suggests that they were deposited by hypogene solutions which may represent either the final ebbing stages of the copper mineralization or an entirely different period of mineralization from an independent source. No minerals have been recognized in the deposit from which uranium could have been derived by weathering processes. If it had been leached from undetectable traces present in the country rock, the metatorbernite would be expected to have more general distribution instead of being concentrated in a relatively small portion of the mineralized area. The absence of an obvious source of uranium is probably not conclusive evidence that metatorbernite was deposited by hypogene solutions, however, no evidence has as yet been found that suggests supergene origin.

Metatorbernite is less widely distributed than wavellite which commonly occurs alone; however, metatorbernite is never found unaccompanied by wavellite. It usually occurs on wavellite crusts and is clearly the younger mineral. Metatorbernite has not been recognized in the leached capping. Most of it has been found in the lower part of the secondary sulfide zone; the maximum depth at which it occurs cannot as yet be determined. In a few places, metatorbernite has been dissolved by supergene solutions leaving a very little residue of a yellowish material resembling kaolinite.

The fact that torbernite transforms to metatorbernite I at 75° C. and to metatorbernite II at 130° C. may indicate that metatorbernite I formed at a temperature between 75° and 130° C. which is certainly above the range of supergene solutions; however, it has not been produced artificially except by dehydration of torbernite, hence there is as yet no proof that metatorbernite I cannot be precipitated from solution at temperatures lower than 75° C.

Jewell Glass⁵ describes an instance in which the inversion of torbernite

⁵ Glass, Jewell J., personal communication.

to metatorbernite I unquestionably took place at room temperature. A specimen of torbernite from Spruce Pine, N. C., stored in a mineral cabinet in the Geological Survey laboratory was reexamined after a lapse of about ten years and was found to have inverted to metatorbernite I. The time required for the inversion to take place at room temperature is not known since no interim examination of the specimen was made; however, it was observed that after a few months, or perhaps years, the emerald-green color of the torbernite had faded to a dull siskin green, and the crystals had lost their transparency.

Very little wavellite has been found in the weathered zone. In a few places in the upper part of the ore body, the wavellite shows evidence of attack by supergene solutions; and some of it is stained by iron oxides; however, much of it occurs in fractures where chalcopyrite is only slightly tarnished by chalcocite and where no other supergene minerals are present. Wavellite commonly crusts pyrite and chalcopyrite, and in one place it was deposited on fresh galena and sphalerite. Many examples of wavellite crusts on barite crystals have been observed.

Wavellite probably forms as a supergene mineral in the weathering of phosphate deposits, but where it occurs in vein deposits it is most likely hypogene, although opinions differ on this point. Lindgren and Ransome⁶ describe hypogene wavellite associated with adularia in the Cripple Creek district. Russell⁷ describes wavellite in the Castle-an-Dinas wolfram mine in Cornwall so as to suggest to the reader that it is a late hydrothermal mineral. The wavellite in the tin veins of Llallagua, Bolivia, is considered hypogene by Turneure⁸ and Bandy,⁹ but Samoyloff¹⁰ and Ahlfeld¹¹ include it among the supergene minerals. In a later discussion of the same tin deposit, Ahlfeld¹² describes wavellite as one of the latest hypogene minerals.

Libethenite crystals are always present on those surfaces of rock fragments to which wulfenite crystals are attached, but many specimens contain libethenite crystals unaccompanied by wulfenite. Both minerals occur in fractured rocks where oxidation and leaching of sulfides are probably complete. The fractures contain abundant limonite, canbyite, malachite, and jarosite. These minerals are clearly younger than the

⁶ Lindgren, W., and Ransome, F. L., *Geology and gold deposits of the Cripple Creek district, Colorado: U. S. Geol. Survey, Prof. Paper* **54**, 176 (1906).

⁷ Russell, Arthur, quoted, Russellite, a new British mineral: *Mineral. Mag.*, **25**, 41 (1938).

⁸ Turneure, F. S., The tin deposits of Llallagua, Bolivia: *Econ. Geol.*, **30**, 60 (1935).

⁹ Bandy, M. C., Direction of flow of mineralizing solutions: *Econ. Geol.*, **37**, 330 (1942).

¹⁰ Samoyloff, V., The Llallagua-Uncia tin deposit: *Econ. Geol.*, **29**, 495 (1934).

¹¹ Ahlfeld, F., The tin ores of Uncia-Llallagua, Bolivia: *Econ. Geol.*, **26**, 253 (1931).

¹² Ahlfeld, F., The tin deposits of Llallagua, Bolivia: *Econ. Geol.*, **31**, 220 (1936).

wulfenite, but their relationship to libethenite is not clear. In some places libethenite crystals are engulfed by them, in other places libethenite crystals appear to have formed on limonite crusts. Clumps of libethenite crystals are commonly attached to wulfenite in such a manner as to suggest that libethenite is decidedly the younger mineral. Thin botryoidal crusts of a black manganiferous mineral resembling pyrolusite are often found on the same rock surfaces with libethenite, which in some examples forms crusts on the manganese mineral. No other occurrence of manganese oxides has been noted in the Castle Dome deposit, hence it is considered to be related to this phase of mineralization. On some rock fragments, barite crystals are attached to the same surfaces as are wulfenite and libethenite, and both minerals appear to have formed on barite crystals.

Although wulfenite and some of the libethenite is clearly older than such definitely supergene minerals as limonite, malachite, canbyite, and jarosite, little evidence can be cited to support a postulation that they were deposited by late hypogene solutions. The usual difficulty in accounting for a source of molybdenum to produce wulfenite by supergene processes is solved in this deposit by the presence of molybdenite, which, although very resistant to oxidation, has been altered to the oxide to some extent; and therefore some molybdenum may have been transported by supergene solutions. Likewise a source of lead is available in galena which is more abundant along the Dome fault system than elsewhere in the deposit. However, Dittler's ¹³ experiments on the synthetic production of wulfenite indicate that an alkalic environment is necessary, and therefore wulfenite is not likely to be formed by supergene solutions. He concludes that it is formed by rising alkaline solutions which reacted with lead carbonate.

Sources of the constituents of libethenite are also readily available in this deposit. The copper could have been derived from hypogene chalcopyrite, and phosphate ion from apatite or wavellite. Lindgren¹⁴ found small crystals of libethenite in cavities and seams in quartzite in the oxidized zone of the Coronado vein in the Clifton-Morenci district. He considered it to be a supergene mineral.

On the basis of field evidence, wulfenite and libethenite are clearly late minerals and may well be of supergene origin; however, wulfenite is earlier than libethenite, and the two minerals are not necessarily genetically related. Their association could easily be coincidental.

¹³ Dittler, E., Further experiments on the synthetic production of wulfenite: *Zeits. Kryst. Min.*, **54**, 332-342 (1914).

¹⁴ Lindgren, W., Copper deposits of the Clifton-Morenci district, Arizona: *U. S. Geol. Survey, Prof. Paper* **43**, 118 (1905).

Turquoise is generally conceded to be a supergene mineral, formed by the action of ground-water solutions, carrying copper and phosphate ions, on kaolin and possibly sericite. Various writers describe all stages in the alteration from copper-stained kaolin to hard blue turquoise; but it is not usually clear from the published descriptions whether the mineral is kaolinite or some other clay. Kaolinite is not a common mineral in the Castle Dome deposit, and its association with turquoise has not been noted; however, turquoise is commonly associated with beidellite, halloysite, and sericite. No evidence has yet been found suggesting that it forms from clay. The hard blue turquoise grades into the soft, white clay-like variety; but the index of refraction of all of it is near 1.62, which is much higher than that of any of the clays present. None of the material could be construed as being transitional between turquoise and the associated clays.

Although turquoise is most abundant in the upper part of the secondary sulfide zone, it is commonly found in deep fractures where chalcopyrite is only slightly replaced by chalcocite or not at all. If turquoise was formed by supergene solutions, the phosphate ion would have to be derived from either wavellite or apatite which are the only earlier phosphate minerals. Wavellite is apparently dissolved by supergene solutions, but much turquoise occurs in parts of the mine where no wavellite has been recognized. In the Castle-an-Dinas mine in Cornwall,¹⁵ wavellite spheres are commonly covered by minute botryoidal aggregates of bright-green turquoise, but no such association has been seen in the Castle Dome deposit. Most writers look to apatite as the probable source of phosphate ion even though admitting in some cases that the country rock contains very little apatite.

¹⁵ Hey, M. H., and Bannister, F. A., Russellite, a new British mineral: *Mineral. Mag.*, 25, 41 (1938).

NOTES AND NEWS

TWINNED PSEUDOMORPHS AFTER PYRITE FROM KING'S BUTTE, GREENE COUNTY, MISSOURI*

MILLER W. ELLIS, *U. S. Geological Survey, Washington, D. C.*

An unusual form of twinned crystals, goethite pseudomorphs after pyrite, has been found in a layer in the Compton limestone of lower Mississippian (Kinderhook) age at King's Butte, Missouri. So far as the writer is aware these complex "paddle-wheel" twins have not heretofore been described. Because of the increasing interest in these twinned crystals shown by mineralogists and collectors, it seems worthwhile to describe their occurrence and physical properties as mineralogical oddities.

King's Butte is a small but conspicuous turretlike rock formation crowning a rounded hill, situated $1\frac{1}{4}$ miles south of North Dry Sac Creek and three-fourths of a mile east of the Prestley Hill cut on State Highway No. 13; it lies 3 miles south of Brighton, and 14 miles north of Springfield, Missouri. The butte proper is capped by an indurated layer of vermicular siltstone (Northview sandstone) underlain by about 40 feet of softer shaly siltstone belonging to the same member; the siltstones form the steep slopes. The more gently rounded hill below the butte is composed of dolomitic limestone of lower Ordovician age. This is overlain by a thin sandstone of lower Mississippian age, which in turn is overlain by several feet of white to buff-colored Compton limestone.

King's Butte has long been known as a good collecting ground for limonitized fossils. The fossils have been preserved as internal and external molds of iron oxide derived from the alteration of iron sulfide. The profusion of limonite fossils, as well as pseudomorphs after the cube and pyritohedron, found on the slopes of the butte bears evidence of the great abundance of pyrite that had been deposited in most of the rocks of the area.

The unusual twinned pseudomorphs occur only on that part of the slope underlain by the limestone formation, and are found in the greatest abundance in the gullies where the Compton limestone is exposed. Some slabs of the limestone contain the twinned crystals in place, and the dark brown residual clay from the disintegration of the limestone rock contains many good specimens. The same type of twinned pseudomorphs has been noted at several other localities, but always in calcareous rocks.

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This restricted occurrence of a distinctly different type of crystallization of a mineral of common occurrence reflects the effect of some physical or chemical difference in the solutions and the sediments of the particular bed or layer in which these crystals were deposited.

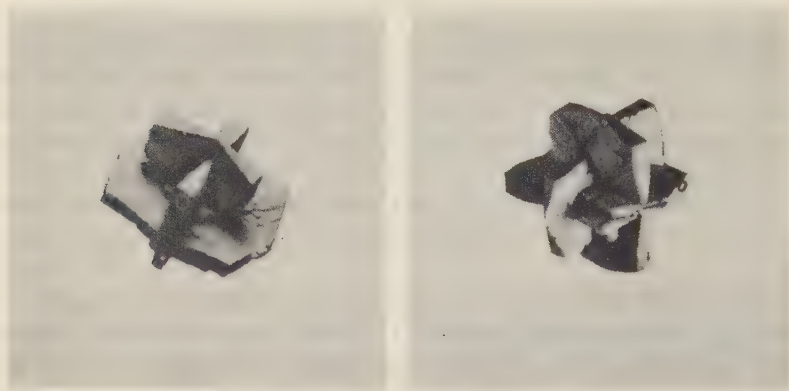


FIG. 1. Twinned pseudomorphs of goethite after pyrite from King's Butte, Greene County, Missouri. The photograph marked with a small white circle is a front view, and the one marked with a black circle is a side view of the same specimen, showing the curious twinning habit and the characteristic striation pattern. (Natural size.)

The two illustrations above are photographs of the same twinned crystal showing the twinning habit and crystallography of a typical specimen. The sizes of the crystals range from 2 millimeters to 20 millimeters. The crystals occur singly and in interlocked groups of 2 to 10 or more. The color ranges from light-brown to nearly black, and the luster varies correspondingly from earthy to submetallic. Some of the crystals are coated by a film of iridescent limonite which gives the surface an apparent but not real metallic luster. Single specimens are composed of three primary, flat, tabular, or platelike crystals, or of three pairs of wedge-shaped crystals, systematically united at right angles to one another. On some specimens secondary plates or wedges have formed adjacent to the primary plate. Striations are common on all of the faces but are best developed on the larger flat surfaces which are distinctly marked by a featherlike pattern having a center rib or joint that corresponds to the shaft of a feather, and a system of fine, slanting striae that correspond to the barbs. The tabular plates are arranged so as to lie in the three planes of symmetry which coincide with the planes of the crystallographic axes and are parallel to the faces of a cube projected on these axes. The striations and traces of lamination on the flat faces of

the plates somewhat resemble those on marcasite; and striations on the ends of the plates resemble those on the rounded faces of a combination of the cube and the pyritohedron. In crystals that do not show apparent distortion, the three axes of symmetry are equal and at right angles to one another. This isometric symmetry is so persistent that it can hardly be accidental.

In order to determine the composition of the pseudomorphs, several of the least altered specimens were crushed and the fragments studied in the Chemical Laboratory of the U. S. Geological Survey. It was found that the residual iron sulfide present in the central core of some of the crystals gave an x -ray diffraction pattern identical with that of pyrite, and the pattern of the reddish-brown iron oxide, the principal part of the pseudomorphs, corresponds to that of goethite.

The author is aware that a goniometric examination of these crystals is certainly in order, and that without such measurements this paper is lacking in its essential requirements, but circumstances do not permit such an examination at this time.

POLYCRASE IN NEW YORK STATE

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Uranium bearing minerals have hitherto been reported from several localities in New York State. E. S. Dana (1892) mentions uranothorite as "From the Champlain iron region, New York, exact locality unknown." Luquer (1896), (1904) and Kerr (1935) have listed and described various uranium bearing minerals from the well known Bedford area. Gratacap (1912) mentions both autunite and torbernite as having been found on "New York Island," presumably Manhattan, and Manchester (1914) states that uraninite was found "years ago" in New York City at Broadway and 155th Street. Pitchblende is reported by Zodac (1939) to have been found in 1924, although not recognized until some years later, associated with molybdenite in thin dikes of grey granite near the east end of the Bear Mountain bridge in Westchester County. Schaub (1940) has identified uraninite in material collected at the Mc-Lear pegmatite near Richville Station, St. Lawrence County.

Other than the lost locality "in the Champlain iron region" no radioactive minerals have been reported from the Precambrian complex of the Adirondack Mountains. Hence the purpose of this paper is to announce a new locality for radioactive minerals in New York State, the

first occurrence of such minerals in the Adirondack Precambrian and the first occurrence of a member of the euxenite-polycrase series.

The mineral now identified as polycrase is found scattered sparsely through a moderately coarse granite pegmatite in the southeast corner of the town of Day, Saratoga County, N. Y. The amount of pegmatite exposed is small; it is therefore, difficult to determine whether it occurs as a lens or as a wide dike. The country rock visible on the south side of the mass is a biotite gneiss striking nearly due east and west with south dips of about 35° . Where the contacts are to be seen the pegmatite

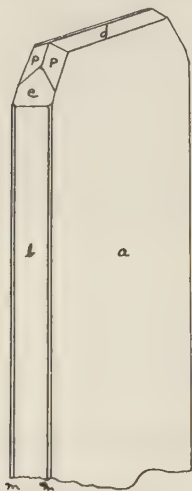


FIG. 1



FIG. 2

appears to be concordantly injected. Attempts to remove feldspar for commercial purposes have resulted in the excavation of two pits along the strike. The openings extend about 250 feet, are from 10 to 30 feet wide and of varying depths. For many years they have been filled with water.

The chief minerals making up the pegmatite are: white microcline, white to buff (rarely pink) orthoclase, colorless to white, or grey to dark smoky brown quartz, with occasional masses of pale rose color. The quartz and orthoclase are often found in medium to coarse grained graphic intergrowths. Biotite is relatively abundant, sometimes in laths a yard or more in length, as is also black tourmaline. The latter mineral has been found in large well terminated crystals associated with the rose quartz masses. Minor constituents are plagioclase, slender apatite crystals, clear greyish green to opaque pink in color, greenish sericite and the polycrase.

The polycrase is commonly found as small orthorhombic crystals enclosed by quartz, feldspar or tourmaline. It appears to be most abundant in association with complexly intergrown masses of black tourmaline. The polycrase crystals are tabular in habit (Fig. 1) prominent forms being pinacoids and domes, less often with prism and pyramid faces. Crystals are usually less than a centimeter in length, but several much larger crystals have been found, one of which measured 27 mm. $\times$ 10 mm. $\times$ 5 mm., another 22 mm. $\times$ 10 mm. $\times$ 4 mm. The crystals when fresh are a dark greenish or brownish black, but, like the polycrase from Henderson County, N. C., they are usually covered with a thin coating of alteration products. The specific gravity of the mineral is about 4.70, the hardness varies from 5.0 to 5.5, cleavage none, fracture small conchoidal, lustre on fresh fracture subvitreous to resinous. The color on fresh fracture is black but in thin splinters is reddish brown because of some light transmission. It is highly radioactive. Admittedly rough tests of the activity by means of a Geiger-Mueller counter indicates about one-tenth that of pitchblende from Spruce Pine, N. C. The radiograph of one of the larger crystals, natural size, was obtained on an Eastman dental x-ray film after 72 hours exposure (Fig. 2).

Through the kindness of the General Electric Company, spectroscopic tests at the Works Laboratory, Schenectady, N. Y., revealed uranium, columbium, yttrium and titanium present, but thorium, cerium, erbium and tantalum absent. These results would indicate the strong probability that the mineral in question is polycrase. Investigations are being continued.

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DEMONSTRATION OF INTERFERENCE FIGURES

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The development of new polarizing materials in the last decades and their current widespread availability in the form of Polaroid have made possible several demonstrations with polarized light that were formerly difficult or impracticable on account of the cost and small aperture of calcite polarizing prisms.

One of the most striking experiments is observation and projection of interference figures by the use of Polaroid plates and polished spheres of birefringent material. The experiment is adapted either for direct study by the student or for screen projection to illustrate a talk. Excellent interference figures may be observed with quartz or corundum spheres $\frac{3}{8}$ to $\frac{1}{2}$ inch in diameter. Calcite and topaz have too much birefringence for observation of color curves, but give fine, sharp isogyres. The experiment is ideal for demonstrating the properties of interference figures, because it lends itself readily to either individual or classroom instruction. The student can see for himself the effect of rotations of the crystal about various axes including that of the rotating stage of the microscope. He can handle the ball himself, observe the position of the uniaxial or the biaxial figure, and determine by experiment the effect of crystal orientation on the interference figure. An effective screen demonstration can be arranged using a vertical projector such as a Spencer Science Delineascope.

Many amateur lapidists are well equipped to cut and at least rough-polish suitable spheres from quartz and other clear crystal. An imperfect polish is sufficient for the experiment. A rough-ground surface may even be "polished" by a coat of clear lacquer. I have recently seen some beautifully polished corundum spheres, diameters from 1 mm. to $\frac{1}{2}$ inch, made by the Laboratory of the Linde Air Products Company at Tona-wanda, N. Y.; these spheres are excellent for the experiment described here. Dr. A. N. Winchell of the American Cyanamid Company, Stamford, Conn., has obtained excellent spheres of calcite and topaz. Each of the materials mentioned gives a result different from that of the others: quartz shows the effect of rotatory polarization; corundum shows the color curves and sometimes slight biaxiality; calcite shows the sharpest isogyres, but almost no color curves can be seen because of their close spacing, due to high birefringence; topaz illustrates the corresponding condition for a biaxial crystal of large axial angle.

This note is published mainly to¹ call the attention of colleagues in the teaching profession to a useful and valuable experiment. No originality

is claimed, though I am not aware of any references describing the demonstration.

PRESERVATION OF SPECIMENS OF MARCASITE AND PYRITE

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The instability of pyrite and marcasite is one that all collectors and curators have experienced, and a cure for which is greatly to be desired. A method of arresting the alteration has been described by Bannister,¹ who also listed localities from which these sulfides were particularly unstable.

The oxidation of pyrite and marcasite results in a series of ferrous sulfates of varying degrees of hydration, but they are hygroscopic and will react with whatever moisture there is in the atmosphere, and the chain reactions gradually cause disintegration of the specimens. Washing with water, in an attempt to dissolve out the salts may produce less soluble basic salts; these are also hygroscopic and the result is merely a postponement of the final breakup of the material.

The following new method has been found successful with pyrite and marcasite specimens which did not contain any associated minerals affected by concentrated HCl; and one must be assured of the absence of even microscopic amounts of such impurities. Pyrite and marcasite are not noticeably attacked by HCl.

The pyrite or marcasite specimen is placed in a closely fitting beaker, and just covered with pure, *colorless, concentrated* HCl. The yellow color quickly assumed by the acid will evince the necessity of the treatment. After a good soaking (perhaps 10 or 15 minutes), the specimen is removed and drained, and placed in fresh, colorless concentrated acid. This procedure is continued until there is no discoloration of the acid. One can now be sure that there are no soluble iron salts left in the pyrite or marcasite, as it takes only a trace of these to discolor the acid.

Since no water must be introduced into the mineral, the specimens are drained, and then soaked in ether until all of the acid is removed. Two soakings in ether should leave the specimens perfectly clean and safe from further alteration even without coating them with plastic films.

Nevertheless, specimens of pyrite and marcasite which have suffered even slight alteration may be quite friable, and it is advisable to soak these in a solution of a plastic to hold them together: the solution recommended by Bannister is one containing 7% (by weight) of vinyl acetate in an equal mixture of acetone and toluene.

¹ Bannister, F. A., *Museums Journal*, **33**, 72-75 (1933); and **36**, 465-476 (1937).

NOTE ON THE ANOMALOUS THERMAL EFFECT IN QUARTZ
OSCILLATOR PLATES

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Fort Collins, Colorado.*

In a recent paper Lukesh and McCaa¹ have reported an anomalous frequency shift in vibrating quartz plates when exposed to infra-red radiation. It is the purpose of this note to confirm and extend these observations. In the last paragraph of their paper Lukesh and McCaa suggest a tentative explanation of the effect in terms of the thermal strains resulting from the differential exposure. Two observations made by the author some time ago tend to confirm their explanation.

When a vibrating *AT* plate is subjected to a mechanical stress in the *X* direction, its frequency changes quite appreciably. For example, compressing a $\frac{1}{2}$ inch 5 mc./sec. *AT* plate along the *X* direction with a force which can be applied directly with the fingers causes a frequency increase of 50 to 100 cycles/sec. The same procedure applied to a *BT* plate causes a decrease in its frequency. The effect in *BT* plates appears to be somewhat less than that in *AT* plates as well as being in the opposite direction. Mechanical stress in the *Z'* direction has very little effect on either plate.

Lukesh and McCaa report that irradiating the central region of the blank while shielding the outer zone produces an increase in the frequency of *AT* plates and a decrease in the frequencies of *BT* plates. These results are easily confirmed. However, if the central portion is shielded while the outer zone is exposed, the effect is reversed. This may easily be demonstrated by shielding the central zone of the vibrating plate with a small metal button which also serves for one of the electrodes.

Very often quartz oscillator plates are clamped between electrodes which also serve as mechanical supports. These electrodes must have a thermal coefficient of expansion which approximates that of quartz in the plane perpendicular to the optic axis. Otherwise an hysteresis effect is observed when the temperature is cycled. For example, in *AT* plates clamped between ceramic electrodes, the frequency is found to rise anomalously when the temperature is increased and to drop anomalously when the temperature is decreased. This phenomenon, which is quite troublesome, is caused by the constraining force exerted on the blank by the electrodes which have a much lower coefficient of expansion than the quartz plate. With *BT* plates the effect is reversed.

All these experimental facts are correlated and explained if we assume

¹ Lukesh, Joseph S., and McCaa, David G., *Am. Mineral.*, **32**, 137-140 (1947).

that exposure of the quartz to infra-red radiation produces localized body heating of the material. Due to the low thermal conductivity of quartz, the expansion resulting from the heating is localized in the exposed region. Thus, exposing the central zone while shielding the outer zone, results in expansion of the central zone against the constraining force supplied by the unexposed outer zone. But lateral compression of the plate is observed to cause a frequency shift in the same sense as that observed when the central zone is irradiated. Hence the hypothesis of Lukesh and McCaa is explained and confirmed.

In our joint paper "Differential thermal analysis of some carbonate minerals," *Am. Mineral.*, **32**, 111-116 (1947), a curve having only an exothermic peak is given for siderite (Fig. 2, p. 114). Since publication several workers in the same field have called to our attention that they most frequently get an endothermic reaction interrupted and followed by the exothermic reaction. Our sample was chosen as the most pure siderite we could obtain. As we point out in paragraph 3, page 115, "The exothermic reaction is a result of the heat balance between the decomposition of the FeCO_3 and the immediate oxidation of the resulting FeO to Fe_2O_3 ." Perhaps a fortuitous combination of pure sample, dilution, and heating rate has suppressed the endothermic reaction which other workers obtain when these conditions are *different*.

F. L. CUTHBERT

RICHARD A. ROWLAND

Dr. A. E. Alexander, director of the Gem Trade Laboratory, Inc., New York, has returned from Bahrain Island, Persian Gulf, where at the invitation of the Bahrain government a study was made of genuine pearl fishing methods as practiced by the natives of that area. On his return several days were spent with Mr. B. W. Anderson, director of the Precious Stone Laboratory, London Chamber of Commerce.

The Crystallographic Society has reported election of the following officers: John W. Gruner, University of Minnesota, president; A. Pabst, University of California, who succeeds to president in 1948, vice-president; William Parrish, Philips Laboratories, Inc., Irvington-on-Hudson, New York, secretary-treasurer (1947-49); and Samuel G. Gordon, Academy of Natural Sciences of Philadelphia, and George Tunnell, University of California at Los Angeles, councilors.

LETTER TO THE EDITOR

X-ray wave-lengths have been expressed in X units. The X unit is defined in terms of the calcite spacing and is nearly 10^{-11} cms., but is now known to differ from 10^{-11} cms. by about 0.2%. During the last twenty-five years X-ray diffraction workers have expressed X-ray wave-lengths and crystal dimensions in terms of a unit which was 1000 X units, but instead of calling it 1000 X units have erroneously called it an Ångström unit. In recent years, the X-ray diffraction groups have agreed to use the term kilo X unit (abbreviated kX) in place of the incorrectly used Ångström unit, until agreement was reached on the best conversion

factor to use for converting from kX to Ångström units. Agreement on the factor has now been reached.

As secretary of the American Society for X-ray and Electron Diffraction, I have been instructed to call the attention of American X-ray workers to the following announcement which appeared in the January, 1947, issue of the *Journal of Scientific Instruments*. Because of its importance it is here reproduced in its entirety.

ELIZABETH ARMSTRONG WOOD, *Secretary*
American Society for X-Ray and Electron
Diffraction
 Bell Telephone Laboratories
 Murray Hill, N. J.

THE CONVERSION FACTOR FOR kX UNITS TO ÅNGSTRÖM UNITS

At the annual conference of the X-ray Analysis Group of the Institute of Physics in July 1946 it was announced that agreement had been reached concerning the factor for converting measurements in kX units to Ångström units. The factor agreed upon, after consultation with the *American Society for X-ray and Electron Diffraction* and Prof. Siegbahn was 1.00202. This factor is probably correct to 0.003%. Since wave-lengths in X-units have been measured to an accuracy of 0.001%, the wave-lengths in Ångström units can be taken as accurate to 0.004% in general.

The following is a list of values of wave-lengths in Ångström units of certain emission lines and absorption edges in common use. The column headed $K\alpha$ gives the mean value of $K\alpha_1$ and $K\alpha_2$, $K\alpha_1$ being allowed twice the weight of $K\alpha_2$.

Current values of the physical constants, such as those quoted by Birge in the 1941 volume of the Physical Society's *Reports on Progress in Physics*, should be used in conjunction with these wave-lengths. In particular density ρ is given by the equation

$$\rho = 1.66020 \Sigma A/V$$

where ΣA is the sum of the atomic weights of the atoms in the unit cell, and V is the volume of the unit cell in $\text{\AA}^3$.

	$K\alpha_1$	$K\alpha_2$	$K\alpha$	$K\beta_1$	Absorption edge
Cr	2.28962	2.29352	2.2909	2.08479	2.0701
Mn	2.10174	2.10570	2.1031	1.91016	1.8954
Fe	1.93597	1.93991	1.9373	1.75654	1.7429
Co	1.78890	1.79279	1.7902	1.62073	1.6072
Ni	1.65783	1.66168	1.6591	1.50008	1.4869
Cu	1.54050	1.54434	1.5418	1.39217	1.3802
Zn	1.43510	1.43894	1.4364	1.29520	1.2831
Mo	0.70926	0.71354	0.7107	0.63225	0.6197
Rh	0.61326	0.61762	0.6147	0.54559	0.5341
Pd	0.58545	0.58982	0.5869	0.52052	0.5090
Ag	0.55941	0.56381	0.5609	0.49701	0.4855

It is recommended that in any published work the values of the wave-lengths used should be explicitly stated.

W. L. BRAGG,
Chairman, X-ray Analysis Group of the
Institute of Physics (England)